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Rev. 07/10/02

VOLUME 4
TERRESTRIAL BIOTA SAMPLING PROJECT PLANS

QUALITY ASSURANCE PROJECT PLAN,
FIELD SAMPLING PLAN, &
SITE-SPECIFIC
HEALTH AND SAFETY PLAN
for the
SITE SAMPLING PLAN

SAUGET AREA 2
(SITES O,P,Q,R,S)
SAUGET, ILLINOIS

May 2001

#2737

Sections

Section 1	Volume 4A	Field Sampling Plan
Section 2	Volume 4B	Quality Assurance Project Plan
Section 3	Volume 4C	Health and Safety Plan

Volume 4A
FIELD SAMPLING PLAN
Terrestrial Biota Sampling
at
SAUGET AREA 2 SITES
(Sites O,P,Q,R,S)
SAUGET, ILLINOIS

May 2001

Prepared for:

Sauget Area 2 Sites Group
c/o Solutia, Inc.
575 Maryville Centre Drive
P.O. Box 66760
St. Louis, Missouri 62141

Prepared by:

AMEC Earth and Environmental, Inc.
285 Davidson Ave., Suite 100
Somerset, NJ 08873
Phone: 732-302-9500

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ACRONYMS

ABRTF	American Bottoms Regional Wastewater Treatment Facility
CERCLA	Comprehensive Environmental Responsibility Compensation and Liability Act
COC	chain-of-custody
ERA	ecological risk assessment
FSP	field sampling plan
HASP	health and safety plan
IEPA	Illinois Environmental Protection Agency
MS/MSD	matrix spike/matrix spike duplicate
PCBs	polychlorinated biphenyls
QAPP	quality assurance project plan
QA/QC	quality assurance/quality compliance
RI/FS	remedial investigation/feasibility study
RPM	regional project manager
SVOC	semi-volatile organic compound
TAL	target analyte list
USEPA	United States Environmental Protection Agency

1.0 PROJECT DESCRIPTION

In accordance with an Administrative Order by Consent between the United States Environmental Protection Agency (USEPA) and the Respondents for the Sauget Area 2 Site, a Remedial Investigation and Feasibility Study (RI/FS) is required for the site. The following Field Sampling Plan (FSP) describes the organization, objectives, and planned sampling and analysis activities to obtain biota samples in support of the terrestrial portion of the Ecological Risk Assessment (ERA) for the Sauget Area 2 Sites located in the Villages of Sauget and Cahokia, St. Clair County, Illinois. The Ecological Risk Assessment is part of the environmental investigations carried out under the RI/FS under the direction of the USEPA Region 5 and the Illinois Environmental Protection Agency (IEPA). Further site details and investigations are described in the RI/FS Support Sampling Plan and Ecological Risk Assessment Workplan.

1.1 INTRODUCTION

This FSP has been prepared for the Sauget Area 2 Sites Group by AMEC Earth and Environmental, Inc. (AMEC) under an Administrative Order by Consent pursuant to Sections 104, 106(a), 107 and 122 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980 (42 USC§§9604, 9606(a), 9607, and 9622.

The Terrestrial and Surface Water/Sediment/Aquatic Biota Quality Assurance Project Plans (QAPP), the Health and Safety Plan (HASP), the Ecological Risk Assessment Workplan, and this FSP define the overall Workplan for the environmental investigation in support of the ERA at the Sites.

1.2 SITE FACILITY DESCRIPTION, HISTORICAL DATA AND CURRENT STATUS

Sauget Area 2 is located in the City of East St. Louis and the Villages of Sauget and Cahokia in St. Clair County, Illinois. The Sauget Area 2 study area is east of the Mississippi River and south of the MacArthur bridge railroad tracks. The study area is west of Route 3 (Mississippi Avenue) and north of Cargill Road.

Site	Former Use	Municipality
Site O	Sewage Sludge Dewatering	Village of Sauget
Site P	Municipal and Industrial Waste Disposal	City of East St. Louis Village of Sauget
Site Q	Municipal and Industrial Waste Disposal	Village of Sauget Village of Cahokia
Site R	Industrial Waste Disposal	Village of Sauget
Site S	Chemical Reprocessing Waste Disposal	Village of Sauget

These sites are located in an area historically used for heavy industry, including chemical manufacturing, metal refining and power generation, and waste disposal. Currently the area is used for heavy industry, warehousing, bulk storage (coal, refined petroleum, lawn and garden products and grain), waste water treatment, hazardous waste treatment, waste recycling and truck terminals. Four commercial establishments are located at the north end of the study area. No residences are located within the study area. Residential areas closest to Sauget Area 2 are approximately 3,000 feet east of Site P and about 3,000 feet east of Site O. These residential areas are located, respectively, in East St. Louis and Cahokia.

1.2.1 Site Location and Physical Setting

Sauget Area 2 is located in the floodplain of the Mississippi River in an area known as American Bottoms. Topographically, the area consists primarily of flat bottom land although local topographic irregularities do occur. Generally, land surface in the American Bottoms slopes from north to south and from east to west toward the Mississippi River. Land surface elevation ranges from 400 to 410 feet above Mean Sea Level (MSL) with little topographic relief.

Sauget Area 2 consists of five former disposal areas, Sites O, P, Q, R and S, adjacent, or in close proximity, to the Mississippi River. These five Sites were given letter designations by the Illinois Environmental Protection Agency (IEPA) in the 1980s. Two of these sites, Sites Q and R, are located on the wet side of the floodwall and levee which is operated and maintained by the US Corps of Engineers and the Metro East Sanitary District. The floodwall is designed to protect the City of East St. Louis and the Villages of Sauget and Cahokia from flooding. Sites O, P and S are located on the dry side of the floodwall and levee.

1.2.2 Present and Past Facility Operations and Disposal Practices

Each of the five sites in Sauget Area 2 is described below. Maximum chemical concentrations included in these site descriptions were included by USEPA in the AOC and are summarized in Table 1.

1.2.2.1 Site O

Site O, located on Mobile Avenue in Sauget, Illinois, occupies approximately 20 acres of land to the northeast of the American Bottoms Regional Wastewater Treatment Facility (ABRTF). An access road to the ABRTF runs through the middle of the site. In 1952, the Village of Sauget Waste Water Treatment Plant began operation at this location. In addition to providing treatment for the Village of Sauget, the plant treated effluent from the various Sauget industries.

During its operation the treatment plant received and treated industrial and municipal wastewater. Approximately 10 million gallons per day of wastewater were treated most of which was from area industries.

Four lagoons were constructed at the wastewater treatment plant in 1965 and placed in operation in 1966/1967. Between 1966/67 and approximately 1978, these lagoons were used to dispose of clarifier sludge from the wastewater treatment plant. They were designated as Site O during a site investigation conducted by IEPA in the 1980s. The lagoons were closed in 1980 by stabilizing the sludge with lime and covering it with approximately two feet of clay. Currently, the lagoons are covered with clay and are vegetated.

Parties that USEPA claims discharged to the Sauget Wastewater Treatment Plant during the time period that the sludge lagoons were in operation included, at a minimum:

- Amax Zinc Corporation,
- American Zinc Company
- Cerro Copper Products Company
- Clayton Chemical Co.
- Darling Fertilizer
- Ethyl Corporation
- Ethyl Petroleum Additives, Inc.
- Midwest Rubber Reclaiming
- Mobil Oil Corporation
- Monsanto Company
- Rogers Cartage Company
- Wiese Planning and Engineering

Parties which own and/or operate, or previously owned and/or operated, portions of Site O include:

- Village of Sauget

The USEPA reports that soil samples collected from Site O contain VOCs, SVOCs, PCBs, dioxin and metals at concentrations of up to:

VOCs (ppb)

Benzene	30,769
Chlorobenzene	58,974
Ethylbenzene	166,667
4-Methyl-2-Pentanone	7,692
Toluene	29,487
Xylenes	615,385

PCBs (ppb)

Aroclor 1232	30,366
Aroclor 1242	1,871,795

Dioxin (ppb)

Tetrachlorodibenzo-p-dioxin	170

Metals (ppm)

Cadmium	31
Copper	341
Mercury	6.3
Nickel	136
Zinc	1,398

SVOCs (ppb)

1,2-Dichlorobenzene	606,000
1,3-Dichlorobenzene	112,821
1,4-Dichlorobenzene	1,030,000
1,2,4-Trichlorobenzene	65,300
1,1,1-Trichloroethane	1,410
1,2,4-Trichlorophenol	26,923
Pentachlorophenol	1,620,000
Benzo(a)anthracene	121,795
Chrysene	282,051
Fluoranthene	74,000
Naphthalene	34,615
Phenanthrene	230,000
Pyrene	282,051
2-Methylnaphthalene	160,256
n-Nitrosodiphenylamine	50,000
Butyl Benzyl Phthalate	3,846,154

The USEPA reports that groundwater samples collected from Site O contain VOCs, SVOCs and metals at concentrations of up to:

VOCs (ppb)	
Benzene	190,000
2-Butanone	62,000
Chlorobenzene	180,000
trans-1,2-Dichloroethene	14,000
Methylene Chloride	52,000
4-Methyl-2-Pentanone	38,000
1,1,2,2-Tetrachoroethane	12,000
Tetrachloroethene	10,000
Toluene	15,000
Trichloroethene	83,000

SVOCs (ppb)	
4-Chloroaniline	780
1,2-Dichlorobenzene	11,000
1,4-Dichlorobenzene	15,000
4-Methylphenol	1,100
Phenol	1,100

Metals (ppb)	
Arsenic	113
Cadmium	11
Lead	6,350

1.2.2.2 Site P

Site P, which is bounded by the Illinois Central Gulf Railroad tracks, the Terminal Railroad Association tracks and Monsanto Avenue, occupies approximately 20 acres of land located in the City of East St. Louis and the Village of Sauget. It was operated by Sauget and Company as an IEPA-permitted landfill from 1973 to approximately 1984 accepting general wastes, including diatomaceous earth filter cake, from Edwin Cooper (now Ethyl Corporation) and non-chemical wastes from Monsanto. IEPA inspections documented the presence of drums labeled "Monsanto ACL-85, Chlorine Composition," drums labeled phosphorus pentasulfide from Monsanto and Monsanto ACL filter residues and packaging. Site P is currently inactive and partially covered, however, access to the site is not restricted.

Parties which USEPA claims to have generated, disposed of, released into and/or transported wastes to Site P include:

- Edwin Cooper Petroleum Additive
- Monsanto Chemical Company

USEPA claims that parties who potentially own, previously owned and/or operated Site P include:

- Chicago Title & Trust Company
- Gulf-Mobile & Ohio Railroad
- Metro East Sanitary District
- Sauget and Company
- Southern Railway System
- Union Electric Company

USEPA reports that soil samples collected from Site P contain VOCs, SVOCs and metals at concentrations of up to:

VOCs (ppb)	
Toluene	413
Xylenes	450

Metals and Inorgancis (ppm)	
Lead	526
Mercury	3.9
Cyanide	15

SVOCs (ppb)	
1,2-Dichlorobenzene	3,625
1,4-Dichlorobenzene	8,875
Phenol	3,875
Di-n-Butylphthalate	16,250

1.2.2.3 Site Q

Site Q, a former subsurface and surface disposal area, occupies approximately 90 acres in the Villages of Sauget and Cahokia. This Site is divided by the Alton and Southern Railroad into a northern portion and a southern portion. The northern portion consists of 65 acres bordered on the north by Site R and Monsanto Avenue. The northern portion is bordered on the south by the main track of the Alton and Southern Railroad and property owned by Patgood Inc. On the east, the northern portion of the site is bordered by the Illinois Gulf Central Railroad and the US Army Corps of Engineers (USACE) flood control levee and on the west the Site is bordered by the Mississippi River.

The southern portion consists of 25 acres, north of Cargill Road and south of the Alton and Southern Railroad. The southern portion is bounded on the west by a ten-foot wide strip of property owned by Union Electric for transmission lines and a spur track of the Alton and Southern Railroad to the Fox Terminal. A barge terminal operated by St. Louis Grain Company is located between the Union Electric property, the spur track and the Mississippi River.

Southern Site Q is bordered on the east by the Illinois Central Gulf Railroad and the flood control levee.

Disposal started in the 1950s and continued until the 1970s. Sauget and Company started operation of a landfill south of the River Terminal in 1966 and terminated operations in 1973. This facility took various wastes including municipal waste, septic tank pumpings, drums, organic and inorganic wastes, solvents, pesticides and paint sludges. It also took plant trash from Monsanto, waste from other industrial facilities and demolition debris.

Most of Site Q is covered with highly permeable black cinders. Eagle Marine Industries and Peavy Company, a division of Con-Agra, operate barge terminal facilities in the central part of the northern portion of Site Q. The southern portion of Site Q is used for reclaiming rebar from concrete and for construction debris disposal. A ten-acre site on the northern portion of Site Q is currently used by Rivercity Landscape Supply as a bulk storage terminal for lawn and garden products. Raw landscape products such as mulch, rock and soil are process and packaged are also processed and packed on this portion of the site.

Access to some portions of the site is restricted by fencing and gates. Other parts of the site have unrestricted access.

Site Q is on the west side of the USCOE floodwall. In 1993, during the highest recorded flood in St. Louis' history, Site Q was flooded. USEPA conducted a CERCLA removal action at the northern portion of Site Q in 1995. USEPA conducted a second CERCLA removal action at the southern portion of Site Q beginning in October of 1999 and into early 2000. During this removal action, USEPA excavated over 2000 drums and over 7,000 cubic yards of contaminated soils containing metals, PCBs, and organics. Excavated material was transported by rail to Oklahoma for disposal at Safety Kleen's Lone Elk hazardous waste landfill.

USEPA claims that the following parties potentially generated, disposed of, released into and/or transported wastes to Site Q;

- AALCO Wrecking Company, Inc.
- Abco Trash Service
- Able Sewer Service
- Ajax Hickman Hauling
- Edgemont Construction
- Edwin Cooper Inc.
- Eight & Trendy Metal Company
- Evans Brothers

- Atlas Service Company
- Banjo Iron Company
- Barry Weinmiller Steel Fabrication
- Becker Iron & Metal Corporation
- Belleville Concrete Cont. Company
- Bi-State Parks Airport
- Bi-State Transit Company
- Boyer Sanitation Service
- Browning-Ferris Industries of St. Louis
- C&E Hauling
- Cargill Inc.
- Century Electric Company
- Circle Packing Company
- Clayton Chemical Company
- Corkery Fuel Company
- Crown Cork & Seal Company, Inc.
- David Hauling
- Dennis Chemical Company, Inc.
- Disposal Service Company
- Dore Wrecking Company
- Dotson Disposal "All" Service
- Dow Chemical
- Finer Metals Company
- Fish Disposal
- Fruin-Colnon Corporation
- Gibson Hauling
- H.C. Fournie Inc.
- H.C. Fournie Plaster
- Hilltop Hauling
- Huffmeier Brothers
- Hunter Packing Company
- Illinois Department of Transportation
- Inmont Corporation
- Lefton Iron & Metal Company
- Mallinckrodt Chemical
- Midwest Sanitation
- Mississippi Valley Control
- Monsanto Company
- Myco-Gloss
- Obear Nestor
- Roy Baur
- Thomas Byrd
- Trash Men Inc.
- United Technologies Corporation
- U.S. Paint Corporation

USEPA claims that the following parties potentially own, previously owned, and/or operated Site Q include:

- Cahokia Trust Properties
- ConAgra, Inc. (leassee)
- Eagle Marine Industries Inc.
- Industrial Salvage & Disposal Company
- Peavey Company
- Pillsbury Company (leasee)
- Sauget & Company
- Union Electric Company
- Village of Cahokia
- Village of Sauget

- Phillips Pipe Line Company

Soil samples collected from Site Q are reported to contain VOCs, SVOCs, metals, PCBs and dioxin. at concentrations of up to:

VOCs (ppb)	
Chlorobenzene	100,000
Ethylbenzene	790,000
4-Methyl-2-Pentanone	250,000
Toluene	2,400,000
Xylenes	2,300,000

PCBs (ppb)	
Aroclor 1248	70,000
Aroclor 1254	360,000
Aroclor 1260	16,000,000

Dioxin (ppb)	
2,3,7,8-TCDD	3.31

SVOCs (ppb)	
1,2-Dichlorobenzene	3,625
1,4-dichlorobenzene	1,200,000
Bis(2-ethylhexyl)phthalate	1,100,000
Di-n-Butylphthalate	900,000

Metals (ppm)	
Antimony	17,900
Arsenic	0.216
Cadmium	152,000
Chromium	3,650
Copper	1,630
Lead	195,000
Mercury	4.9
Nickel	371
Selenium	59.5
Silver	30.2
Thallium	0.89
Zinc	9,520

Groundwater samples collected from Site Q contain VOCs, SVOCs, metals and Inorganics at concentrations of up to:

VOCs (ppb)

Benzene	2,000
Chlorobenzene	6,700
1,2-Dichloroethane	3,000
2-Hexanone	3,500
4-Methyl-2-pentanone	2,700
Toluene	1,600

Metals and Inorganics (ppm)

Arsenic	0.100
Cyanide	1,560

SVOCs (ppb)

4-Chloroaniline	15,000
Phenol	190,000
2-Chlorophenol	33,000
2, 4-Dichlorophenol	14,000
2,4,6-Trichlorophenol	6,000
Pentachlorophenol	35,000
4-Methylphenol	23,000
2,4-Dimethylphenol	2,800
2-Nitroaniline	2,000
Acenaphthylene	3,900

1.2.2.4 Site R

Site R, a closed industrial-waste disposal area owned by Solutia Inc, is located between the flood control levee and the Mississippi River in Sauget, Illinois. Its northern border is Monsanto Avenue and its southern border is Site Q. A portion of Site Q, known as the "Dog Leg", is located to the east of Site R. This site once called the "Sauget Toxic Dump" and the "Monsanto Landfill" it is now known as the "River's Edge Landfill".

Industrial Salvage and Disposal, Inc. (ISD) operated the River's Edge Landfill for Monsanto from 1957 to 1977. Hazardous and non-hazardous bulk liquid and solid chemical wastes and drummed chemical wastes from Monsanto's W.G. Krummrich plant and, to a lesser degree, its Queeny plant in St. Louis were disposed at Site R. Disposal began in the northern portion of the site and expanded southward. Wastes contained phenols, aromatic nitro compounds, aromatic amines, aromatic nitroamines, chlorinated aromatic hydrocarbons, aromatic and aliphatic carboxylic acids and condensation products of these compounds.

In 1979, Monsanto completed the installation of a clay cover on Site R to cover waste, limit infiltration through the landfill, and prevent direct contact with fill material. The cover's thickness ranges from two feet to approximately eight feet. In 1985, Monsanto installed a 2,250-foot long rock revetment along the east bank of the Mississippi River adjacent to Site R. The purpose of the stabilization project was to prevent further erosion of the riverbank and thereby minimize potential for the surficial release of waste material from the landfill. During the 1993 flood, Site

R was flooded but the clay cap was not overtopped. No erosion of the river bank or cap resulted from this flood.

Access to Site R is restricted by fencing and is monitored by plant personnel.

On February 13, 1992, the State of Illinois and Monsanto signed a consent decree entered in St. Clair County Circuit Court requiring further remedial investigations and feasibility studies to be conducted by Monsanto on Site R. The results of the Remedial Investigation/Feasibility Study were submitted to Illinois EPA in 1994. Solutia made a good faith offer to the IEPA to install an engineered cap and a leachate recovery system in 1997.

Parties who are claimed to own, previously have owned, and/or operated Site R include:

- Cahokia Trust Properties
- Monsanto Company
- Solutia Inc
- Sauget and Company

Sediment samples collected from a drainage ditch around Site R showed VOC concentrations ranging from 0.002 to 0.035 ppm. SVOC concentrations in sediments ranged from 0.045 to 3.99 ppm. PCBs were detected at concentrations ranging from 0.08 to 1.5 ppm. Elevated levels of metals, particularly aluminum, iron and magnesium, were also detected. Sediment samples collected adjacent to the Mississippi River on the west side of Site R showed SVOC concentrations ranging from 0.001 to 7.7 ppm. PCBs were also detected at concentrations ranging from 0.00001 to 0.23 ppm.

Soil samples collected from Site R showed elevated levels of VOCs ranging from 0.15 to 5,800 ppm. SVOCs were found at levels ranging from 0.017 to 19,000 ppm. Pesticides were found at levels ranging from 0.011 to 99 ppm and PCBs were detected at levels ranging from 0.075 to 4,800 ppm. Elevated levels of arsenic, chromium, lead, nickel and mercury were also detected in Site R soils.

SVOC concentrations in leachate samples ranged from 0.6 to 12.3 ppb. Pesticide concentrations ranged from 0.5 to 3.0 ppb and PCBs were detected at 0.08 ppb. Dioxin/furan concentrations ranged from 0.0001 to 0.0014 ppm. Cyanide was also detected at 71 ppb.

Groundwater samples collected from wells on and immediately downgradient of Site R had VOCs concentrations ranging up to 38,136 ppb. SVOCs were detected at concentrations as high as 2,973,885 ppb. Historical groundwater data is presented in the Site Sampling Plan (SSP). The following constituents were detected at the following maximum concentrations:

Upper Hydrogeologic Unit (UHU)

VOCs (ppb)		SVOCs (ppb)	
Acetone	69,000	Aniline	200,000
Benzene	11,300	2-Chloroaniline	300,000
Bromoform	4,700	3-Chloroaniline	100,000
2-Butanone	3,100	4-Chloroaniline	200,000
Chlorobenzene	158,000	2-Nitroaniline	500,000
Chloroethane	10,000	4-Nitroaniline	500,000
Chloroform	1,600		
1,1-Dichloroethane	4,700	1,2-Dichlorobenzene	100,000
1,2-Dichloroethane	16,500	1,3-Dichlorobenzene	100,000
1,1-Dichloroethene	2,800	1,4-Dichlorobenzene	100,000
trans-1,2-Dichloroethene	11,300	1,2,4-Trichlorobenzene	100,000
Methylene Chloride	22,400		
4-Methyl-2-pentanone	3,100	Nitrobenzene	100,000
1,1,2,2-Tetrachloroethane	7,000	2-Nitrochlorobenzene	3,400,000
Tetrachloroethene	4,100	3-Nitrochlorobenzene	730,000
Toluene	6,000	4-Nitrochlorobenzene	1,500,000
1,1,1-Trichloroethane	3,800		
Trichloroethene	4,610	Phenol	2,000,000
Vinyl Chloride	24,500	2-Chorophenol	540,000
		4-Chlorophenol	210,000
		2,4-Dichlorophenol	340,000
		2,4,6-Trichlorophenol	100,000
		3-Methylphenol	280,000
		4-Methylphenol	47,000
		2,4-Dimethylphenol	100,000

4-Chloro-3-Methylphenol	100,000
4-Nitrophenol	500,000
Naphthalene	100,000
2-Chloronaphthalene	100,000
Benzoic Acid	50,800
Benzyl Alcohol	1,830
Bis(2-chloroethoxy)methane	100,000
Bis(2-ethylhexyl)phthalate	100,000
4-Nitrodiphenylamine	1,250

Middle Hydrogeologic Unit (MHU)

VOCs (ppb)	
Acetone	22,000
Benzene	9,980
Chlorobenzene	60,200
Chloroform	400
Chloromethane	2,500
1,1-Dichloroethane	1,200
1,2-Dichloroethane	9,200
1,1-Dichloroethene	700
trans-1,2-Dichloroethene	11,300
Ethylbenzene	2,500
Methylene Chloride	2,260
4-Methyl-2-Pentanone	3,100
Tetrachloroethene	1,050
Toluene	3,000
1,1,1-Trichloroethane	950
Trichloroethene	500
Vinyl Chloride	2,500
Xylenes	2,500

SVOCs (ppb)	
Aniline	685,000
2-Chloroaniline	329,000
3-Chloroaniline	57,200
4-Chloroaniline	105,000
1,2-Dichlorobenzene	25,000
1,3-Dichlorobenzene	25,000
1,4-Dichlorobenzene	25,000
1,2,4-Trichlorobenzene	25,000
Nitrobenzene	25,000
2-Nitrochlorobenzene	463,000
3-Nitrochlorobenzene	460,000
4-Nitrochlorobenzene	185,000
Phenol	1,100,000
2-Chorophenol	160,000
4-Chlorophenol	67,000
2,4-Dichlorophenol	83,000
2,4,6-Trichlorophenol	25,000

Pentachlorophenol	125,000
3-Methylphenol	110,000
2,4-Dimethylphenol	25,000
4-Nitrophenol	125,000
Naphthalene	25,000
Chrysene	25,000
Fluoranthene	25,000
Pyrene	25,000
n-Nitrosodiphenylamine	25,000
Bis(2-ethylhexyl)phthalate	25,000

Deep Hydrogeologic Unit (DHU)

VOCs (ppb)	
Acetone	500
Benzene	613
Chlorobenzene	7,380
Chloromethane	500
1,2-Dichloroethane	1,910
trans-1,2-Dichloropropene	720
Ethylbenzene	500
Methylene Chloride	1,790
4-Methyl-2-Pentanone	1,190
Tetrachloroethene	1,220
Toluene	2,070
Trichloroethene	500
Xylenes	962

SVOCs (ppb)	
Aniline	48,000
2-Chloroaniline	195,000
3-Chloroaniline	52,400
4-Chloroaniline	56,900
2-Nitroaniline	5,000
4-Nitroaniline	5,000
1,2-Dichlorobenzene	9,810
1,3-Dichlorobenzene	950
1,4-Dichlorobenzene	2,250
1,2,4-Trichlorobenzene	950
Nitrobenzene	1,010
2-Nitrochlorobenzene	219,000
3-Nitrochlorobenzene	30,900
4-Nitrochlorobenzene	115,000
Phenol	33,000
2-Chorophenol	8,500

4-Chlorophenol	18,000
2,4-Dichlorophenol	12,800
2,4,6-Trichlorophenol	3,030
Pentachlorophenol	2,500
2,4-Dimethylphenol	1,400
Benzo(a)pyrene	1,300
Benzo(k)fluoranthene	1,300
Chrysene	1,300
Fluoranthene	1,100
Naphthalene	800
Pyrene	950
4-Nitrodiphenylamine	5,000
n-Nitrosodiphenylamine	1,900
Bis(2-chloroethyl)ether	2,900
Bis(2-cloroisopropyl)ether	2,900
Bis(2-ethylhexyl)phthalate	5,000
Di-n-Butylphthalate	5,000
3,3'-Dichlorobenzidene	8,500
Hexachlorocyclopentadiene	10,000

1.2.2.5 Site S

Site S is located southwest of Site O. Allegedly, the property is, or was, owned by the Village of Sauget and the Resource Recovery Group. In the mid-1960s, solvent recovery began on site under Clayton Chemical, which is now owned by the Resource Recovery Group (RRG). The waste solvents were steam-stripped resulting in still bottoms that were allegedly disposed of in a shallow, on-site excavation that is now designated Site S. Historical aerial photographs indicate that Site S was potentially a waste and/or drum disposal area. The northern portion of the site is grassed and its southern portion is covered with gravel and fenced.

Soil samples collected from Site S are reported to contain VOCs, SVOCs, PCBs and metals at concentrations up to:

VOCs (ppb)	
Ethylbenzene	450,000
4-Methyl-2-Pentanone	93,000
Toluene	990,000
1,1,1-Trichloroethane	12,000
Xylenes	620,000

SVOCs (ppb)	
Naphthalene	200,000
Bis(2-ethylhexyl)phthalate	20,000,000
Butyl Benzyl Phthalate	490,000
Di-n-Butylphthalate	1,500,000
di-n-Octylphthalate	310,000

PCBs (ppb)	
Aroclor 1248	85,000
Aroclor 1254	69,000
Aroclor 1260	41,000

Metals (ppm)	
Copper	139
Lead	0.392
Mercury	3.5
Zinc	327

Additional information regarding environmental investigation at the Sites may be found in the Site Sampling Plan. A summary of the maximum concentrations of constituents detected in soil during the initial soil sampling event is provided in Table 1.

1.3 PROJECT OBJECTIVES AND SCOPE

The purpose of this RI/FS is to gather sufficient information to quantify potential risks to ecological and human health receptors based on the presence of chemical residuals in environmental media. This FSP reflects the objectives of the terrestrial portion of the ERA only, which is to determine if chemicals detected in soil will affect the terrestrial ecosystem.

Vegetation (e.g., grasses) will be collected to characterize the potential for contaminant uptake in plants, and if so, the potential adverse effects to the herbivorous prairie vole (*Microtus ochrogaster*). Because earthworms (*Lumbricus sp.*) ingest soil, these organisms will be collected to characterize potential adverse impacts to a vermivore, the short-tailed shrew (*Blarina brevicauda*). In order to correlate with contaminant concentrations in soil, samples of biota will be taken from the same locations where the soil/waste characterization samples are collected. Samples of biota will be analyzed for chemicals that may bioaccumulate including SVOCs, pesticides, herbicides, PCBs, dioxins/furans, and Target Analyte List (TAL) metals.

Due to sample size limitations for earthworms, sample analyses hierarchy will be performed, if necessary. Earthworm samples will be analyzed for PCBs first, followed by other parameters including SVOCs, dioxins, herbicides, pesticides, lipids, and metals if sufficient sample mass can be collected.

2.0 ECOLOGICAL ASSESSMENT FIELD SAMPLING PLAN

This section describes the conceptual approach for the sampling event, locations for collection of biota samples, collection procedures, numbers of samples to be collected, labeling and chain-of-custody requirements, and sample container and preservation and holding time requirements.

2.1 STUDY AREA

The ecological assessment focuses on Sites O, P, Q, R, and S, as described in Section 1.2.

2.2 FIELD SAMPLING RATIONALE AND SAMPLING LOCATIONS

Four soil and waste characterization samples are proposed for collection in each of Sites O,P,R, and S during other RI/FS field activities. Because of the relative size of Site Q compared to the other Sites, an additional four soil and waste characterization samples will be collected there. The sampling of biota will be co-located with surface soil samples to determine if chemical contamination in soils poses adverse ecological risks to terrestrial biota inhabiting or feeding from the sites. The surface soil samples in all Sites will be located in the worst case locations based on the results of a reconnaissance using electromagnetic induction (EMI) and soil gas survey equipment to be conducted by URS. However, if the worst case locations chosen by URS do not have any habitat for the selected ecological receptors (other than in Level A), the field crew has the option to collect another surface soil sample at the nearest location providing suitable habitat. Soil sampled for the ecological risk assessment should be sieved to remove large pieces of gravel and vegetation which do not contribute directly to either earthworm, plant or wildlife incidental ingestion of soil. The sample will be analyzed for the same parameters as the URS sample, including total organic carbon, and used for the location of the earthworm and plant samples.

The terrestrial portion of the Ecological Assessment Field Sampling Plan consists of two separate field events: a reconnaissance survey and the sampling event.

2.2.1 Reconnaissance Survey Objectives

A reconnaissance survey will be conducted in late August 2001 to refine the field sampling activities proposed in the following sections. The observations made during the survey will be used to provide a verbal and photographic description of the sites, finalize sampling locations, procedures, and the number of biota samples that can be realistically collected during the sampling program. The objectives and justification for the Survey activities are described below.

- Photodocument Sites O, P, Q, R, and S.
- Locate previous soil/waste characterization sample locations from the RI to help determine locations for co-located soil and biota samples from each Site.
- Finalize representative receptor species for use as assessment endpoints.
- Conduct a qualitative fauna/flora survey and habitat and cover type assessments. Direct and indirect (e.g., calls, scat, tracks) observations will be documented.
- Determine the dominant vegetation species and relative abundance for subsequent sampling.
- Determine the most appropriate sampling techniques.

The results of the survey will be summarized in a concise technical memorandum for submission to the EPA. The technical memo will summarize the chemical results of screening sample analysis, present the observations made during the recon survey, and confirm the position to collect terrestrial and aquatic samples during the field work. The qualitative fauna/flora survey and the estimate of the dominant vegetation species and relative abundance of other species will be used to evaluate changes in plant community abundance and diversity across a constituent concentration gradient within each of the Sites. To the extent that historical soil concentration data are available, the gradient over which significant differences in plant community abundance and diversity occur can be used to develop potential reference locations. Highly disturbed Sites such as the semi-permeable cap in Site R can potentially represent reference locations for other comparable areas.

Modifications to the field sampling program as a result of the reconnaissance survey will be documented in an amendment to this Field Sampling Plan and the QAPP. Field work will not be conducted until the USEPA has approved the final Worplan.

2.2.2 Sampling Program

The sampling program is scheduled to begin in October 2001. During the sampling program, biota samples will be collected for laboratory analysis of target compounds. The objectives of the sampling program are as follows:

- Collect vegetation and earthworms in Sites O, P, Q, R, and S for chemical analyses of tissues. Concentrations of target analytes in biotic tissue will be used in dietary exposure models for the selected receptor species for extrapolation to assessment endpoints. Information on tissue analyses will be used to evaluate potential effects on the terrestrial foodchain.

- The objective of vegetation sampling is to determine concentrations of target analytes for use in exposure models for the representative prairie vole; the objective of the earthworm sampling is to determine concentrations of contaminants in earthworms due to the earthworm's ingestion of contaminated soil for use in exposure models for the short-tailed shrews. Both rodents are prey for the carnivorous red fox (*Vulpes vulpes*), the terrestrial receptor. Body burdens in the short-tailed shrews will be estimated using earthworm body burdens and exposure models such as those presented by USEPA (1999).

- Biota tissue samples will be stored in dry ice prior to and for shipment to the laboratory. At the laboratory, biota samples will be stored frozen prior to chemical analysis, as described in Table 2. Frozen storage of tissue samples can be maintained for a maximum of one year, consistent with USEPA guidance on solid and tissue sample preservation (40 CFR 136.3). The method-specified holding times for extraction and analyses begin when samples are thawed for preparation and analysis. Preservation and holding times are listed in Table 2.

2.2.3 Sample Locations

As discussed above, sample locations for both vegetation and earthworms will be from the same locations as soil/waste characterization samples collected as part of the Sauget Area 2 RI/FS. Locations will be verified during the reconnaissance survey.

2.3 VEGETATION SAMPLING

The goal of vegetation sampling is to collect a sufficient amount of vegetative matter from the dominant species for chemical analysis at each of the 24 soil sample locations.

2.3.1 Vegetation Sample Collection

Subsequent to classification of the herbivorous vegetation (e.g., grasses), the dominant species will be determined and sufficient sample (approximately 25 grams per fraction will be required for SVOCs, pesticides, herbicides, PCBs, dioxins, and metals) will be collected to make a composite of 175 to 200 grams for laboratory analysis.

2.3.2 Vegetation Sample Analytes, Containers, and Shipment Requirements

Terrestrial plants will be collected for chemical analysis of tissues to estimate exposure to rodents, such as the prairie vole, that may feed on the vegetation. To minimize variability associated with differential uptake by plant species, an effort will be made to analyze the same or similar plant species at all locations. The selection of species will be guided by observations made during the reconnaissance survey.

Different portions of the plant can be used as a food source; however, for the purposes of this RI/FS, only aboveground portions (stems/leaves/seeds) will be collected for chemical analysis. Vegetation will be collected using decontaminated stainless steel scissors or shears. Each sample will be a composite of enough vegetation to comprise sufficient sample for analysis. The composite will be washed with distilled water to remove soil from the vegetation. While it is readily accepted that dry deposition can account for a significant portion of foliar contaminant levels, the objective of the sampling is to assess the potential for contaminant uptake from the soils to the leaf structures. Dry deposition and particulate resuspension in many ways cannot be directly attributed to site-related activities for which the PRPs are responsible. Therefore, the leaves will be washed with distilled water to remove that confounding factor. The sample will then be placed in a sealable plastic bag and placed on dry ice for shipment to the laboratory for analysis of SVOCs, pesticides, herbicides, PCBs, dioxins, percent moisture, and metals. Each sample will be assigned a unique number and will also be labeled with the data of collection, time, and initials of the collector. Sample analyses, preservation, containers and holding time requirements are provided in Table 2.

2.4 EARTHWORM SAMPLING

The goal of macroinvertebrate sampling is to obtain sufficient earthworms biomass for tissue analysis of chemicals at each of 24 soil sample locations.

2.4.1 Earthworm Sample Collection

Earthworms will be collected by digging through soil using a field decontaminated, stainless steel trowel or shovel, as necessary. The laboratory requires approximately 25 grams of sample per sample fraction (i.e., SVOCs, herbicides, pesticides, PCBs, dioxins, metals, lipids) for a total sample of 175 to 200 grams. Additional sample material will be necessary for matrix QC, including the matrix spike/matrix spike duplicate (MS/MSD) and matrix duplicate. The following scheme will be used to achieve sufficient earthworm sample size within a reasonable time period for the sampling program.

- The sampling team will initially dig a hole to a depth of approximately six inches at the location of the soil sample. Then additional effort will be made to cut around the edges "as quickly and deeply as possible" excavating a hole approximately 25 to 40 cm across and 10 to 30 cm deep (10 to 16 in and 4 to 12 in, respectively) (James, 1996). The root mat will be carefully disaggregated and searched for worms Depending on the amount of earthworms encountered, additional soil will be removed from the hole until enough worms have been collected.

- After collection is completed, samples will be washed and rinsed with distilled water, placed in a sealable plastic bag, and stored on dry ice for shipment to the laboratory.

2.4.2 Earthworm Sample Analytes, Containers, and Shipment Requirements

Earthworms will be collected for chemical analysis of tissues to estimate exposure to vermivores, such as the short-tailed shrew. Each sample will be a composite of enough earthworms to allow for chemical analysis of all parameters, if possible. The composite will be washed to remove soil from the outside of the earthworms[1] then placed in a sealable plastic bag and placed on dry ice for shipment to the laboratory for analysis of SVOCs, pesticides, herbicides, PCBs, dioxins, lipids, and metals. Each sample will be assigned a unique number and will also be labeled with the data of collection, time, and initials of the collector. Sample analyses, preservation, containers and holding time requirements are provided in Table 2.

2.5 SAMPLE NUMBERING

Each sample will have its own unique number to designate the contractor (AMEC), the specific Site where the sample is collected from (i.e., O, P, Q, R, S), the medium being sample [i.e., surface water (SW), sediment (SED), omnivorous fish (OF), carnivorous fish (CF), plants (P), and earthworm (W)], the sequential sample number, and the date. Thus, bottles will be labeled:

AMEC-SiteP-P-1-020701

Bottle labels will also have the following information: sampler's initials, time collected, and analytical parameters (e.g., PCBs), and preservatives, as necessary.

[1] Earthworms will not be depurated prior to chemical analysis to simulate actual conditions.

3.0 SAMPLE CUSTODY

Chain-of-custody (COC) procedures for biota collection will follow custody protocols as described in USEPA guidance[2].

3.1 Field Chain of Custody Procedures

The sample packaging and shipment procedures summarized below will ensure that samples will arrive under proper chain-of-custody.

(a) The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. The number of persons handling the samples should be kept to a minimum to preserve sample integrity.

(b) All bottles will be identified with unique sample numbers and locations on secure bottle labels that will include sample identification numbers, location, date of collection, time of collection, and type of analysis required.

(c) Sample labels will be marked with waterproof ink.

(d) Samples will be accompanied with a properly completed COC form that will include sample numbers and locations. Further field custody documentation and transfer procedures are described in subsequent sections.

3.2 Field Logbooks/Documentation

Field logbooks will provide the means of recording data collection activities. As such, entries will be described in as much detail as possible so that persons subsequently entering the site may reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook

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[2] USEPA, 1985. NEIC Policies and Procedures, EPA-330/9-78DDI-R.

with be identified by the project-specific document number. The title page of each logbook will contain the following:

- person to whom the logbook is assigned.
- logbook number
- project name
- project start date, and
- project end date.

Entries made at the beginning of each entry will include the date, start time, weather, names of all sampling team members present and their affiliation, level of personal protection being used, and the signature of the person making the entry. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will also be recorded in the field logbook.

Measurements made, photographs taken, and samples collected will be recorded. All entries must be made in black ink. No erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark and initialed and dated by the person correcting it. Whenever a sample is collected or a measurement is made, a detailed description of the station location, including compass and distance measurements, shall be recorded. The number of the photographs taken of the location will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

The procedures and equipment used to collect samples will be noted along with the time of sampling, sample description, depth at which the sample was collected (as applicable), and amount and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples will receive a unique sampling number and will also be recorded in the field logbook.

3.1.3 Transfer of Custody and Shipment Procedures

The sample packaging and shipment procedures summarized below will ensure that samples will arrive with proper COC.

(a) Samples are accompanied by a properly completed COC form that will include sample numbers and locations. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and record the time on the COC. The COC documents the transfer of sample custody from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

(b) Samples will be properly packaged for shipment, including ice to preserve all samples at $\leq 4^{\circ}C$ and dispatched to the appropriate laboratory for analysis, with a separate, signed COC form enclosed in each shipping container. Shipping containers will be secured with strapping tape and custody seals will be affixed prior to shipment to the laboratory.

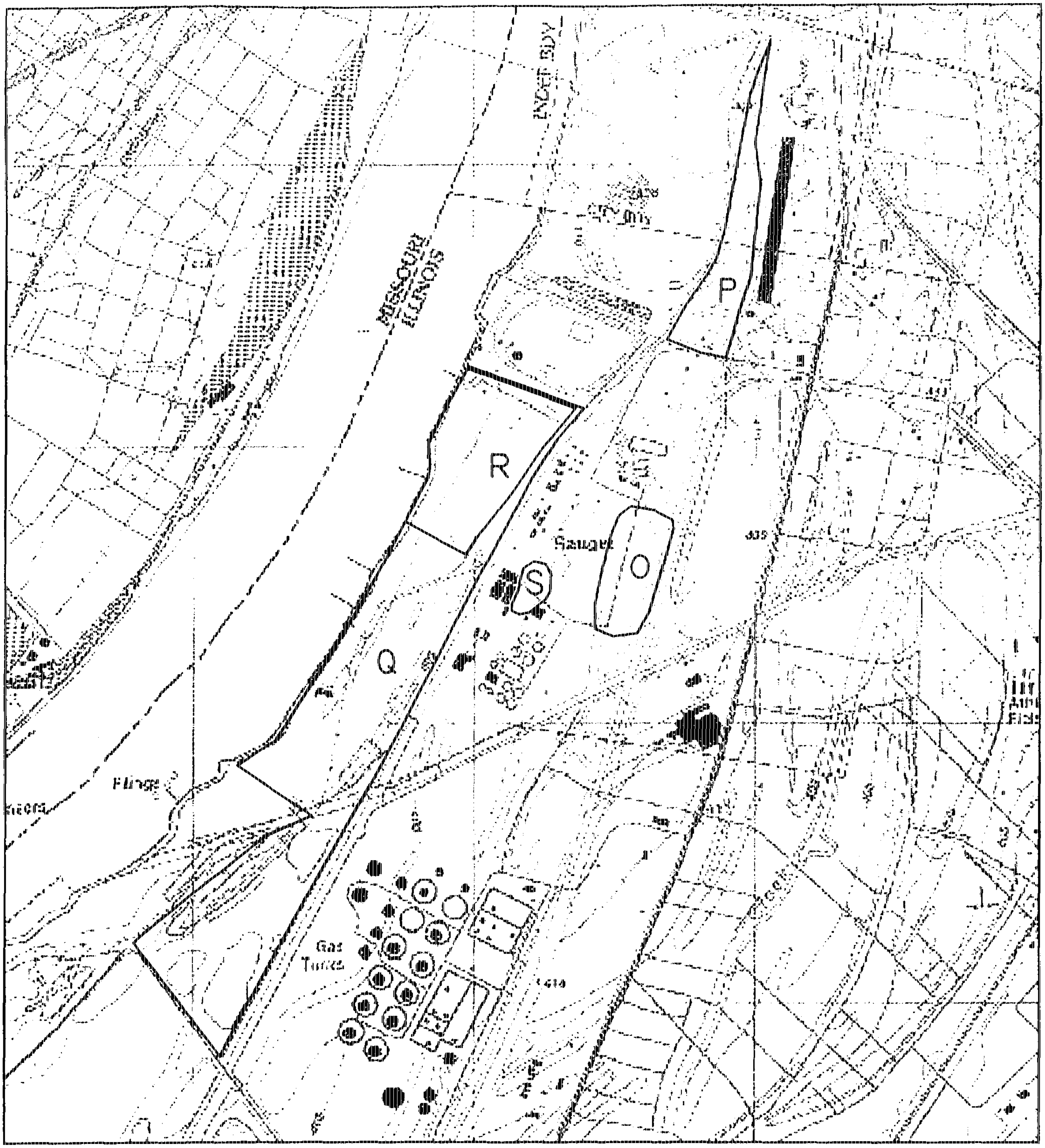
(c) All shipments will be accompanied by the COC record identifying the contents. The original record will accompany the shipment and copies of the COC will be retained by the field personnel for documentation. It is recommended that a copy of the COC be faxed to the laboratory on the date of collection.

(d) If the samples are sent by common carrier, a bill of lading (e.g., airbill) should be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody form as long as the custody forms remain sealed inside the sample cooler and the custody seals remain intact.

4.0 REFERENCES

James, S., 1996. Earthworms. *In*: Methods for the Examination of Organismal Diversity in Soils and Sediments. (G. Hall, ed.). CAB Internat., New York. pp. 249-262.

USEPA, 1999. Screening Level Ecological Risk Assessment at Hazardous Waste Combustion Facilities, Vol. 1, 2, & 3. Peer Review Draft. August 1999. EPA530-D-99-001A, EPA530-D-99-001B, and EPA530-D-99-001C.



SOURCE: USGS QUADRANGLE (CAHOKIA, IL-MO), 1998
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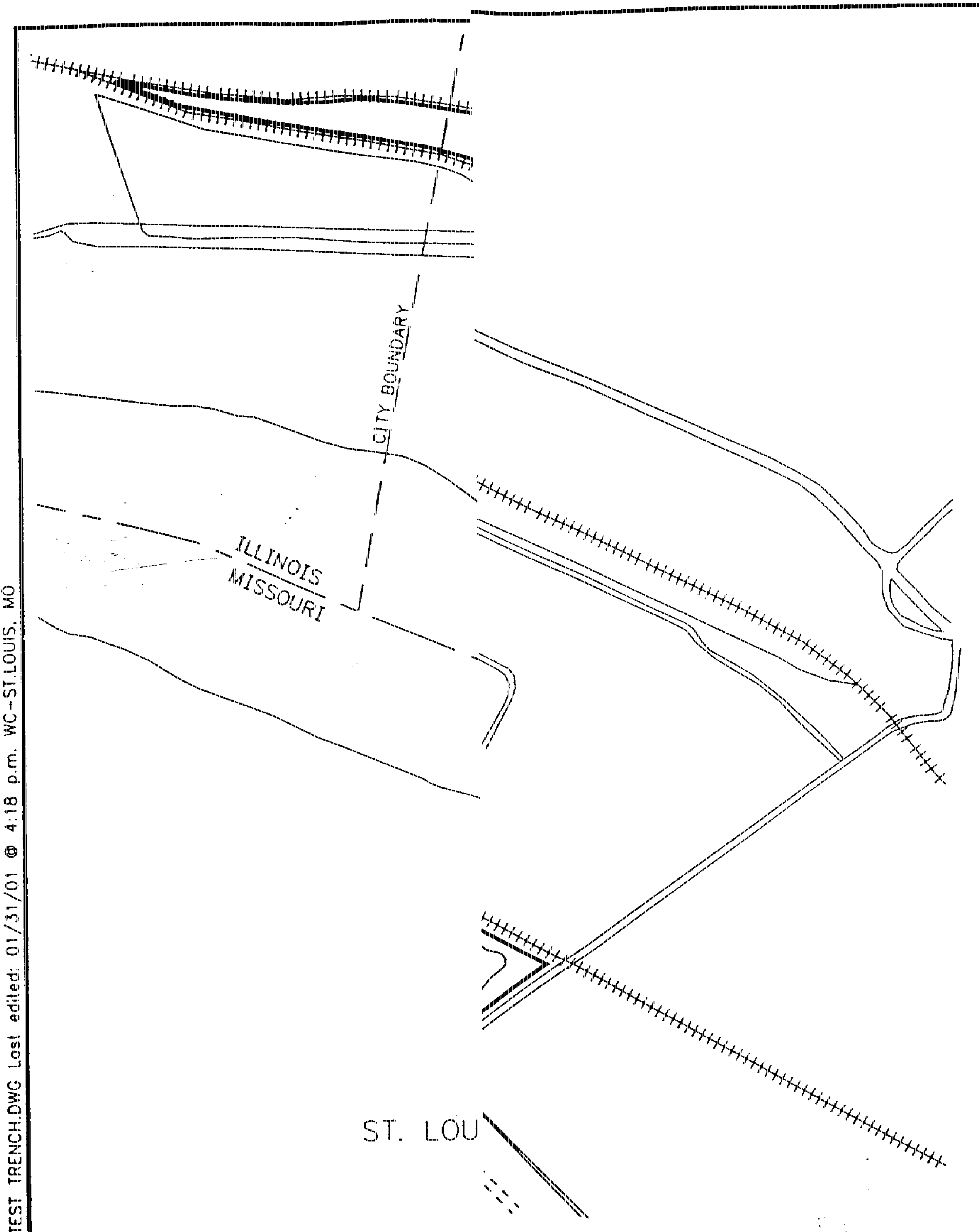
FIGURE 1

SITE LOCATION MAP

SAUGET AREA 2
SITES O, P Q, R, S
SAUGET AND CAHOKIA, ILLINOIS



. 2 RI/FS SAMPLING LOCATIONS IOIS	PROJECT NO. 2320010024.00

1/01	Site Vicinity Map	FIG. NO. 1

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Table 1.1
Maxiumum Constituent Concentrations in Soil
Sauget Area 2

Constituent	Site O	Site P	Site Q	Site R [A]	Site S
VOCs				5800	
benzene	30.8				
chlorobenzene	58.9		100		
ethylbenzene	167		790		450
4-methyl-2-pentanone	7.69		250		93
toluene	29.5	0.413	2400		990
1,1,1-trichloroethane	1.41				12
o-xylene			2300		
xylenes(total)	615.4	0.45			620
SVOCs				19000	
1,4-dichlorobenzene	1030	8.87	1200		
1,2-dichlorobenzene	606	3.625			
1,2,4-trichlorophenol	26.9				
naphthalene	34.6				200
2-methylnapthalene	160				
N-nitrosodiphenylamine	50				
pentachlorophenol	1620				
phenanthrene	230				
fluoranthene	74				
pyrene	282				
butyl benzyl phthalate	3846				490
benzo(a)anthracene	121.7				
1,2,4-trichloroebenzene	65.3				
chrysene	282				
phenol		3.875			
di-n-butyl-phthalate		16.25	900		1500
di-n-octyl-phthalate					310
bis(2-ethylhexyl)phthalate			1100		20000
PCBs				4800	
Aroclor 1232	30.3				
Aroclor 1242	1871				
Aroclor 1248			70		85
Aroclor 1254			360		69
Aroclor 1260			16000		41
Dioxins					
2,3,7,8-TCDD	0.00017		0.0033		
Metals					
antimony			17900		
arsenic			0.216		
cadmium	31		152000		
chromium			3650		
copper	341		1630		139
cyanide		15			
lead		526	195000		0.392
mercury	6.3	3.9	4.9		3.5
nickel	136		371		
selenium			59.9		
silver			30.2		
thallium			0.89		
zinc	1398		9520		327

all concentrations in ppm

[A] - Soil sampling at Site R showed VOC concentrations ranging from .15 to 5800 ppm. SVOCs were found at levels ranging from 0.017 to 19,000 ppm. Pesticides were found at levels ranging from 0.11 to 99 ppm and PCBs were detected at levels ranging from 0.75 to 4,800 ppm. Elevated levels of arsenic, chromium, lead, nickel, and mercury were also detected in Site R soils.

Table 2

Biota Tissue Analyses: Analytical Method, Number, Sample Preservation, Container Specifications, and Holding Time Requirements

Parameter	Analytical Method[a]	Number of Samples [b]	QA/QC Samples			Sample Container	Preservative	Holding Time [c]
			Field Blanks	Duplicates [c]	MS/MSD			
SVOCs	8240	24 vegetation/ 24 earthworm	5	2	1	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
Herbicides	8151A	24 vegetation/ 24 earthworm	5	2	1	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
Pesticides	8081A	24 vegetation/ 24 earthworm	5	2	1	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
Dioxins/ Dibenzofurans	8090[d]	24 vegetation/ 24 earthworm	5	2	1	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
PCBs	680	24 vegetation/ 24 earthworm	5	2	1	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
Metals	6010B (ICP)/7000 Series Methods (GFAA)/mercury - 7471A	24 vegetation/ 24 earthworm	5	2	1	sealable plastic bag	Store at <-10°C (dry ice)	180 days - all metals except mercury. Mercury - 28 days.

(a) - Analytical Method is from USEPA SW-846, 3rd Edition, December 1996 unless otherwise specified

(b) - Number of samples does not include QA/QC samples.

(c) - IEPA requires one duplicate per 10 samples collected

(d) - All holding times start from the time samples are thawed, if received frozen

(e) - SW-846, 3rd Edition, September 1994 method included in the December 1996 update

VOLUME 4B
TERRESTRIAL BIOTA
QUALITY ASSURANCE PROJECT PLAN
SAUGET AREA 2 SITES
(Sites O,P,Q,R,S)
SAUGET, ILLINOIS

May 2001

Prepared for:

Sauget Area 2 Sites Group
c/o Solutia, Inc.
575 Maryville Centre Drive
P.O. Box 66760
St. Louis, Missouri 62141

Prepared by:

AMEC Earth and Environmental, Inc.
285 Davidson Ave., Suite 100
Somerset, NJ 08873
Phone: 732-302-9500

VOLUME 4B

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Phone: 732-302-9500

Steven Smith, Site Program Manager Chairman, Sauget Area 2 Sites Group Solutia, Inc.	Date:
Charles Harman, Ecological Project Manager AMEC Earth and Environmental, Inc.	Date:
Candy Morin, Project Manager Illinois EPA	Date:
Kristin McCracken, Regional QA Manager Severn Trent Laboratories - Savannah (STL)	Date:
Greg Johnson, QA Manager Triangle Laboratories	Date
Michael McAteer, USEPA Project Manager USEPA Region 5	Date:

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ATTACHMENTS

LIST OF ACRONYMS

ABRTF	American Bottoms Regional Wastewater Treatment Facility
CCB	continuing calibration blank
CERCLA	Comprehensive Environmental Responsibility Compensation and Liability Act
CLP	contract laboratory program
COC	chain-of-custody
CV	calibration verification
DQO	data quality objective
ERA	ecological risk assessment
FSP	field sampling plan
GC/MS	gas chromatograph/mass spectroscopy
GPC	gel permeation chromatography
HASP	health and safety plan
ICB	initial calibration blank
ICP	inductively coupled plasma
IEPA	Illinois Environmental Protection Agency
LCS	laboratory control sample
MD	matrix duplicate
MDL	method detection limit
MS/MSD	matrix spike/matrix spike duplicate
NIST	National Institute of Standards and Technology
PCBs	polychlorinated biphenyls
QAPP	quality assurance project plan
QA/QC	quality assurance/quality compliance
RBCs	risk-based concentrations
RI/FS	remedial investigation/feasibility study
RL	reporting limit
RPD	relative percent difference
RPM	regional project manager
SOPs	standard operating procedures
SRM	standard reference material
SVOC	semi-volatile organic compound
TAL	target analyte list

TIC	tentatively identified compounds
USEPA	United States Environmental Protection Agency
VOCs	volatile organic compounds

1.0 PROJECT DESCRIPTION

In accordance with an Administrative Order by Consent between the USEPA and the Respondents for the Sauget Area 2 Site, a Remedial Investigation and Feasibility Study (RI/FS) is required for the site. The following Quality Assurance Project Plan (QAPP) describes the organization, objectives, planned sampling and analysis activities, and the specific quality assurance/quality control (QA/QC) procedures to generate valid and usable data from biota samples in support of the Ecological Risk Assessment (ERA) for the Sauget Area 2 Sites located in the Villages of Sauget and Cahokia, St. Clair County, Illinois. The Ecological Risk Assessment is part of the environmental investigations carried out under the RI/FS under the direction of the USEPA Region 5 and the Illinois Environmental Protection Agency (IEPA). Further site details and investigations are described in the RI/FS Support Sampling Plan and Ecological Risk Assessment Workplan.

1.1 INTRODUCTION

The Field Sampling Plan (FSP), the Health and Safety Plan (HASP), the Ecological Risk Assessment Workplan, and Surface Water/Sediment/Aquatic Biota QAPP define the overall Workplan for the environmental investigation in support of the ERA at the Sites. This QAPP includes the FSP for the terrestrial ERA sampling activities; the FSP will also be submitted as a separate document.

1.2 SITE FACILITY DESCRIPTION, HISTORICAL DATA AND CURRENT STATUS

Sauget Area 2 is located in the City of East St. Louis and the Villages of Sauget and Cahokia in St. Clair County, Illinois. The Sauget Area 2 study area is east of the Mississippi River and south of the MacArthur bridge railroad tracks. The study area is west of Route 3 (Mississippi Avenue) and north of Cargill Road.

Site	Former Use	Municipality
Site O	Sewage Sludge Dewatering	Village of Sauget
Site P	Municipal and Industrial Waste Disposal	City of East St. Louis Village of Sauget
Site Q	Municipal and Industrial Waste Disposal	Village of Sauget Village of Cahokia

Site R	Industrial Waste Disposal	Village of Sauget
Site S	Chemical Reprocessing Waste Disposal	Village of Sauget

These sites are located in an area historically used for heavy industry, including chemical manufacturing, metal refining and power generation, and waste disposal. Currently, the area is used for heavy industry, warehousing, bulk storage (coal, refined petroleum, lawn and garden products and grain), waste water treatment, hazardous waste treatment, waste recycling and truck terminals. Four commercial establishments are located at the north end of the study area. No residences are located within the study area. Residential areas closest to Sauget Area 2 are approximately 3,000 feet east of Site P and about 3,000 feet east of Site O. These residential areas are located, respectively, in East St. Louis and Cahokia.

1.2.1 Site Location and Physical Setting

Sauget Area 2 is located in the floodplain of the Mississippi River in an area known as American Bottoms. Topographically, the area consists primarily of flat bottom land although local topographic irregularities do occur. Generally, land surface in the American Bottoms slopes from north to south and from east to west toward the Mississippi River. Land surface elevation ranges from 400 to 410 feet above Mean Sea Level (MSL) with little topographic relief.

Sauget Area 2 consists of five former disposal areas, Sites O, P, Q, R and S, adjacent, or in close proximity, to the Mississippi River. These five Sites were given letter designations by the Illinois Environmental Protection Agency (IEPA) in the 1980s. Two of these sites, Sites Q and R, are located on the wet side of the floodwall and levee which is operated and maintained by the US Corps of Engineers and the Metro East Sanitary District. The floodwall is designed to protect the City of East St. Louis and the Villages of Sauget and Cahokia from flooding. Sites O, P and S are located on the dry side of the floodwall and levee.

1.2.2 Present and Past Facility Operations and Disposal Practices

Each of the five sites in Sauget Area 2 is described below. Maximum chemical concentrations included in these site descriptions were included by USEPA in the AOC and are summarized in Table 1.

1.2.2.1 Site O

Site O, located on Mobile Avenue in Sauget, Illinois, occupies approximately 20 acres of land to the northeast of the American Bottoms Regional Wastewater Treatment Facility (ABRTF). An access road to the ABRTF runs through the middle of the site. In 1952, the Village of Sauget Waste Water Treatment Plant began operation at this location. In addition to providing treatment for the Village of Sauget, the plant treated effluent from the various Sauget industries.

During its operation, the treatment plant received and treated industrial and municipal wastewater, most of which was from area industries, at a rate of approximately 10 million gallons per day.

Four lagoons were constructed at the wastewater treatment plant in 1965 and placed in operation in 1966/1967. Between 1966/67 and approximately 1978, these lagoons were used to dispose of clarifier sludge from the wastewater treatment plant. They were designated as Site O during a site investigation conducted by IEPA in the 1980s. The lagoons were closed in 1980 by stabilizing the sludge with lime and covering it with approximately two feet of clay. Currently, the lagoons are covered with clay and are vegetated.

Parties that USEPA claims discharged to the Sauget Wastewater Treatment Plant during the time period that the sludge lagoons were in operation included, at a minimum:

- Amax Zinc Corporation,
- American Zinc Company
- Cerro Copper Products Company
- Clayton Chemical Co.
- Darling Fertilizer
- Ethyl Corporation
- Ethyl Petroleum Additives, Inc.
- Midwest Rubber Reclaiming
- Mobil Oil Corporation
- Monsanto Company
- Rogers Cartage Company
- Wiese Planning and Engineering

Parties which own and/or operate, or previously owned and/or operated, portions of Site O include:

- Village of Sauget

The USEPA reports that soil samples collected from Site O contain VOCs, SVOCs, PCBs, dioxin and metals at concentrations of up to:

VOCs (ppb)

Benzene	30,769
Chlorobenzene	58,974
Ethylbenzene	166,667
4-Methyl-2-Pentanone	7,692
Toluene	29,487
Xylenes	615,385

PCBs (ppb)

Aroclor 1232	30,366
Aroclor 1242	1,871,795

Dioxin (ppb)

Tetrachlorodibenzo-p-dioxin	170

Metals (ppm)

Cadmium	31
Copper	341
Mercury	6.3
Nickel	136
Zinc	1,398

SVOCs (ppb)

1,2-Dichlorobenzene	606,000
1,3-Dichlorobenzene	112,821
1,4-Dichlorobenzene	1,030,000
1,2,4-Trichlorobenzene	65,300
1,1,1-Trichloroethane	1,410
1,2,4-Trichlorophenol	26,923
Pentachlorophenol	1,620,000
Benzo(a)anthracene	121,795
Chrysene	282,051
Fluoranthene	74,000
Naphthalene	34,615
Phenanthrene	230,000
Pyrene	282,051
2-Methylnaphthalene	160,256
n-Nitrosodiphenylamine	50,000
Butyl Benzyl Phthalate	3,846,154

The USEPA reports that groundwater samples collected from Site O contain VOCs, SVOCs and metals at concentrations of up to:

VOCs (ppb)	
Benzene	190,000
2-Butanone	62,000
Chlorobenzene	180,000
trans-1,2-Dichloroethene	14,000
Methylene Chloride	52,000
4-Methyl-2-Pentanone	38,000
1,1,2,2-Tetrachoroethane	12,000
Tetrachloroethene	10,000
Toluene	15,000
Trichloroethene	83,000

SVOCs (ppb)	
4-Chloroaniline	780
1,2-Dichlorobenzene	11,000
1,4-Dichlorobenzene	15,000
4-Methylphenol	1,100
Phenol	1,100

Metals (ppb)	
Arsenic	113
Cadmium	11
Lead	6,350

1.2.2.2 Site P

Site P, which is bounded by the Illinois Central Gulf Railroad tracks, the Terminal Railroad Association tracks and Monsanto Avenue, occupies approximately 20 acres of land located in the City of East St. Louis and the Village of Sauget. It was operated by Sauget and Company as an IEPA-permitted landfill from 1973 to approximately 1984, accepting general wastes, including diatomaceous earth filter cake, from Edwin Cooper (now Ethyl Corporation) and non-chemical wastes from Monsanto. IEPA inspections documented the presence of drums labeled "Monsanto ACL-85, Chlorine Composition," drums labeled phosphorus pentasulfide from Monsanto and Monsanto ACL filter residues and packaging. Site P is currently inactive and partially covered, however, access to the site is not restricted.

Parties which USEPA claims to have generated, disposed of, released into and/or transported wastes to Site P include:

- Edwin Cooper Petroleum Additive
- Monsanto Chemical Company

USEPA claims that parties who potentially own, previously owned and/or operated Site P include:

- Chicago Title & Trust Company
- Sauget and Company
- Gulf-Mobile & Ohio Railroad
- Southern Railway System
- Metro East Sanitary District
- Union Electric Company

USEPA reports that soil samples collected from Site P contain VOCs, SVOCs and metals at concentrations of up to:

VOCs (ppb)	
Toluene	413
Xylenes	450

SVOCs (ppb)	
1,2-Dichlorobenzene	3,625
1,4-Dichlorobenzene	8,875
Phenol	3,875
Di-n-Butylphthalate	16,250

Metals and Inorgancis (ppm)	
Lead	526
Mercury	3.9
Cyanide	15

1.2.2.3 Site Q

Site Q, a former subsurface and surface disposal area, occupies approximately 90 acres in the Villages of Sauget and Cahokia. This Site is divided by the Alton and Southern Railroad into a northern portion and a southern portion. The northern portion consists of 65 acres bordered on the north by Site R and Monsanto Avenue. The northern portion is bordered on the south by the main track of the Alton and Southern Railroad and property owned by Patgood, Inc. On the east, the northern portion of the site is bordered by the Illinois Gulf Central Railroad and the US Army Corps of Engineers (USACE) flood control levee and on the west the Site is bordered by the Mississippi River.

The southern portion consists of 25 acres, north of Cargill Road and south of the Alton and Southern Railroad. The southern portion is bounded on the west by a ten-foot wide strip of property owned by Union Electric for transmission lines and a spur track of the Alton and Southern Railroad to the Fox Terminal. A barge terminal operated by St. Louis Grain Company is located between the Union Electric property, the spur track and the Mississippi River.

Southern Site Q is bordered on the east by the Illinois Central Gulf Railroad and the flood control levee.

Disposal started in the 1950s and continued until the 1970s. Sauget and Company started operation of a landfill south of the River Terminal in 1966 and terminated operations in 1973. This facility took various wastes including municipal waste, septic tank pumpings, drums, organic and inorganic wastes, solvents, pesticides and paint sludges. It also took plant trash from Monsanto, waste from other industrial facilities, and demolition debris.

Most of Site Q is covered with highly permeable black cinders. Eagle Marine Industries and Peavy Company, a division of Con-Agra, operate barge terminal facilities in the central part of the northern portion of Site Q. The southern portion of Site Q is used for reclaiming rebar from concrete and for construction debris disposal. A ten-acre site on the northern portion of Site Q is currently used by Rivercity Landscape Supply as a bulk storage terminal for lawn and garden products. Raw landscape products such as mulch, rock and soil are process and packaged are also processed and packed on this portion of the site.

Access to some portions of the site is restricted by fencing and gates. Other parts of the site have unrestricted access.

Site Q is on the west side of the USCOE floodwall. In 1993, during the highest recorded flood in St. Louis' history, Site Q was flooded. USEPA conducted a CERCLA removal action at the northern portion of Site Q in 1995. USEPA conducted a second CERCLA removal action at the southern portion of Site Q beginning in October of 1999 and into early 2000. During this removal action, USEPA excavated over 2000 drums and over 7,000 cubic yards of contaminated soils containing metals, PCBs, and organics. Excavated material was transported by rail to Oklahoma for disposal at Safety Kleen's Lone Elk hazardous waste landfill.

USEPA claims that the following parties potentially generated, disposed of, released into and/or transported wastes to Site Q;

- AALCO Wrecking Company, Inc.
- Abco Trash Service
- Able Sewer Service
- Edgemont Construction
- Edwin Cooper Inc.
- Eight & Trendy Metal Company

- Ajax Hickman Hauling
- Atlas Service Company
- Banjo Iron Company
- Barry Weinmiller Steel Fabrication
- Becker Iron & Metal Corporation
- Belleville Concrete Cont. Company
- Bi-State Parks Airport
- Bi-State Transit Company
- Boyer Sanitation Service
- Browning-Ferris Industries of St. Louis
- C&E Hauling
- Cargill Inc.
- Century Electric Company
- Circle Packing Company
- Clayton Chemical Company
- Corkery Fuel Company
- Crown Cork & Seal Company, Inc.
- David Hauling
- Dennis Chemical Company, Inc.
- Disposal Service Company
- Dore Wrecking Company
- Dotson Disposal "All" Service
- Dow Chemical
- Evans Brothers
- Finer Metals Company
- Fish Disposal
- Fruin-Colnon Corporation
- Gibson Hauling
- H.C. Fournie Inc.
- H.C. Fournie Plaster
- Hilltop Hauling
- Huffmeier Brothers
- Hunter Packing Company
- Illinois Department of Transportation
- Inmont Corporation
- Lefton Iron & Metal Company
- Mallinckrodt Chemical
- Midwest Sanitation
- Mississippi Valley Control
- Monsanto Company
- Myco-Gloss
- Obear Nestor
- Roy Baur
- Thomas Byrd
- Trash Men Inc.
- United Technologies Corporation
- U.S. Paint Corporation

USEPA claims that the following parties potentially own, previously owned, and/or operated Site Q include:

- Cahokia Trust Properties
- ConAgra, Inc. (lessee)
- Eagle Marine Industries Inc.
- Pillsbury Company (lessee)
- Sauget & Company
- Union Electric Company

- Industrial Salvage & Disposal Company
- Peavey Company
- Phillips Pipe Line Company
- Village of Cahokia
- Village of Sauget

Soil samples collected from Site Q are reported to contain VOCs, SVOCs, metals, PCBs and dioxin. at concentrations of up to:

VOCs (ppb)	
Chlorobenzene	100,000
Ethylbenzene	790,000
4-Methyl-2-Pentanone	250,000
Toluene	2,400,000
Xylenes	2,300,000

PCBs (ppb)	
Aroclor 1248	70,000
Aroclor 1254	360,000
Aroclor 1260	16,000,000

Dioxin (ppb)	
2,3,7,8-TCDD	3.31

SVOCs (ppb)	
1,2-Dichlorobenzene	3,625
1,4-dichlorobenzene	1,200,000
Bis(2-ethylhexyl)phthalate	1,100,000
Di-n-Butylphthalate	900,000

Metals (ppm)	
Antimony	17,900
Arsenic	0.216
Cadmium	152,000
Chromium	3,650
Copper	1,630
Lead	195,000
Mercury	4.9
Nickel	371
Selenium	59.5
Silver	30.2
Thallium	0.89
Zinc	9,520

Groundwater samples collected from Site Q contain VOCs, SVOCs, metals and Inorganics at concentrations of up to:

VOCs (ppb)		SVOCs (ppb)	
Benzene	2,000	4-Chloroaniline	15,000
Chlorobenzene	6,700	Phenol	190,000
1,2-Dichloroethane	3,000	2-Chlorophenol	33,000
2-Hexanone	3,500	2, 4-Dichlorophenol	14,000
4-Methyl-2-pentanone	2,700	2,4,6-Trichlorophenol	6,000
Toluene	1,600	Pentachlorophenol	35,000
		4-Methylphenol	23,000
Metals and Inorganics (ppm)		2,4-Dimethylphenol	2,800
Arsenic	0.100	2-Nitroaniline	2,000
Cyanide	1,560	Acenaphthylene	3,900

1.2.2.4 Site R

Site R, a closed industrial-waste disposal area owned by Solutia Inc, is located between the flood control levee and the Mississippi River in Sauget, Illinois. Its northern border is Monsanto Avenue and its southern border is Site Q. A portion of Site Q, known as the "Dog Leg", is located to the east of Site R. This site was once called the "Sauget Toxic Dump" and the "Monsanto Landfill"; it is now known as the "River's Edge Landfill".

Industrial Salvage and Disposal, Inc. (ISD) operated the River's Edge Landfill for Monsanto from 1957 to 1977. Hazardous and non-hazardous bulk liquid and solid chemical wastes and drummed chemical wastes from Monsanto's W.G. Krummrich plant and, to a lesser degree, its Queeny plant in St. Louis were disposed at Site R. Disposal began in the northern portion of the site and expanded southward. Wastes contained phenols, aromatic nitro compounds, aromatic amines, aromatic nitroamines, chlorinated aromatic hydrocarbons, aromatic and aliphatic carboxylic acids and condensation products of these compounds.

In 1979, Monsanto completed the installation of a clay cover on Site R to cover waste, limit infiltration through the landfill, and prevent direct contact with fill material. The cover's thickness ranges from two feet to approximately eight feet. In 1985, Monsanto installed a 2,250-foot long rock revetment along the east bank of the Mississippi River adjacent to Site R. The purpose of the stabilization project was to prevent further erosion of the riverbank and thereby minimize potential for the surficial release of waste material from the landfill. During the 1993 flood, Site R was flooded but the clay cap was not overtopped. No erosion of the river bank or cap resulted from this flood.

Access to Site R is restricted by fencing and is monitored by plant personnel.

On February 13, 1992, the State of Illinois and Monsanto signed a consent decree entered in St. Clair County Circuit Court requiring further remedial investigations and feasibility studies to be conducted by Monsanto on Site R. The results of the Remedial Investigation/Feasibility Study were submitted to Illinois EPA in 1994. Solutia made a good faith offer to the IEPA to install an engineered cap and a leachate recovery system in 1997.

Parties who are claimed to own, previously have owned and/or operated Site R include:

- Cahokia Trust Properties
- Monsanto Company
- Solutia Inc
- Sauget and Company

Sediment samples collected from a drainage ditch around Site R showed VOC concentrations ranging from 0.002 to 0.035 ppm. SVOC concentrations in sediments ranged from 0.045 to 3.99 ppm. PCBs were detected at concentrations ranging from 0.08 to 1.5 ppm. Elevated levels of metals, particularly aluminum, iron and magnesium, were also detected. Sediment samples collected adjacent to the Mississippi River on the west side of Site R showed SVOC concentrations ranging from 0.001 to 7.7 ppm. PCBs were also detected at concentrations ranging from 0.00001 to 0.23 ppm.

Soil samples collected from Site R showed elevated levels of VOCs ranging from 0.15 to 5,800 ppm. SVOCs were found at levels ranging from 0.017 to 19,000 ppm. Pesticides were found at levels ranging from 0.011 to 99 ppm and PCBs were detected at levels ranging from 0.075 to 4,800 ppm. Elevated levels of arsenic, chromium, lead, nickel and mercury were also detected in Site R soils.

SVOC concentrations in leachate samples ranged from 0.6 to 12.3 ppb. Pesticide concentrations ranged from 0.5 to 3.0 ppb and PCBs were detected at 0.08 ppb. Dioxin/furan concentrations ranged from 0.0001 to 0.0014 ppm. Cyanide was also detected at 71 ppb.

Groundwater samples collected from wells on and immediately downgradient of Site R had VOCs concentrations ranging up to 38,136 ppb. SVOCs were detected at concentrations as high as 2,973,885 ppb. Historical groundwater data is presented in the Site Sampling Plan (SSP). The following constituents were detected at the following maximum concentrations:

Upper Hydrogeologic Unit (UHU)

VOCs (ppb)		SVOCs (ppb)	
Acetone	69,000	Aniline	200,000
Benzene	11,300	2-Chloroaniline	300,000
Bromoform	4,700	3-Chloroaniline	100,000
2-Butanone	3,100	4-Chloroaniline	200,000
Chlorobenzene	158,000	2-Nitroaniline	500,000
Chloroethane	10,000	4-Nitroaniline	500,000
Chloroform	1,600		
1,1-Dichloroethane	4,700	1,2-Dichlorobenzene	100,000
1,2-Dichloroethane	16,500	1,3-Dichlorobenzene	100,000
1,1-Dichloroethene	2,800	1,4-Dichlorobenzene	100,000
trans-1,2-Dichloroethene	11,300	1,2,4-Trichlorobenzene	100,000
Methylene Chloride	22,400		
4-Methyl-2-pentanone	3,100	Nitrobenzene	100,000
1,1,2,2-Tetrachloroethane	7,000	2-Nitrochlorobenzene	3,400,000
Tetrachloroethene	4,100	3-Nitrochlorobenzene	730,000
Toluene	6,000	4-Nitrochlorobenzene	1,500,000
1,1,1-Trichloroethane	3,800		
Trichloroethene	4,610	Phenol	2,000,000
Vinyl Chloride	24,500	2-Chorophenol	540,000
		4-Chlorophenol	210,000

2,4-Dichlorophenol	340,000
2,4,6-Trichlorophenol	100,000
3-Methylphenol	280,000
4-Methylphenol	47,000
2,4-Dimethylphenol	100,000
4-Chloro-3-Methylphenol	100,000
4-Nitrophenol	500,000
Naphthalene	100,000
2-Chloronaphthalene	100,000
Benzoic Acid	50,800
Benzyl Alcohol	1,830
Bis(2-chloroethoxy)methane	100,000
Bis(2-ethylhexyl)phthalate	100,000
4-Nitrodiphenylamine	1,250

Middle Hydrogeologic Unit (MHU)

VOCs (ppb)	
Acetone	22,000
Benzene	9,980
Chlorobenzene	60,200
Chloroform	400
Chloromethane	2,500
1,1-Dichloroethane	1,200
1,2-Dichloroethane	9,200
1,1-Dichloroethene	700
trans-1,2-Dichloroethene	11,300
Ethylbenzene	2,500
Methylene Chloride	2,260
4-Methyl-2-Pentanone	3,100
Tetrachloroethene	1,050
Toluene	3,000

SVOCs (ppb)	
Aniline	685,000
2-Chloroaniline	329,000
3-Chloroaniline	57,200
4-Chloroaniline	105,000
1,2-Dichlorobenzene	25,000
1,3-Dichlorobenzene	25,000
1,4-Dichlorobenzene	25,000
1,2,4-Trichlorobenzene	25,000
Nitrobenzene	25,000
2-Nitrochlorobenzene	463,000
3-Nitrochlorobenzene	460,000
4-Nitrochlorobenzene	185,000

1,1,1-Trichloroethane	950
Trichloroethene	500
Vinyl Chloride	2,500
Xylenes	2,500

Phenol	1,100,000
2-Chorophenol	160,000
4-Chlorophenol	67,000
2,4-Dichlorophenol	83,000
2,4,6-Trichlorophenol	25,000
Pentachlorophenol	125,000
3-Methylphenol	110,000
2,4-Dimethylphenol	25,000
4-Nitrophenol	125,000
Naphthalene	25,000
Chrysene	25,000
Fluoranthene	25,000
Pyrene	25,000
n-Nitrosodiphenylamine	25,000
Bis(2-ethylhexyl)phthalate	25,000

Deep Hydrogeologic Unit (DHU)

VOCs (ppb)	
Acetone	500
Benzene	613
Chlorobenzene	7,380
Chloromethane	500
1,2-Dichloroethane	1,910
trans-1,2-Dichloropropene	720
Ethylbenzene	500
Methylene Chloride	1,790
4-Methyl-2-Pentanone	1,190
Tetrachloroethene	1,220
Toluene	2,070
Trichloroethene	500

SVOCs (ppb)	
Aniline	48,000
2-Chloroaniline	195,000
3-Chloroaniline	52,400
4-Chloroaniline	56,900
2-Nitroaniline	5,000
4-Nitroaniline	5,000
1,2-Dichlorobenzene	9,810
1,3-Dichlorobenzene	950
1,4-Dichlorobenzene	2,250
1,2,4-Trichlorobenzene	950

Xylenes	962	Nitrobenzene	1,010
		2-Nitrochlorobenzene	219,000
		3-Nitrochlorobenzene	30,900
		4-Nitrochlorobenzene	115,000
		Phenol	33,000
		2-Chorophenol	8,500
		4-Chlorophenol	18,000
		2,4-Dichlorophenol	12,800
		2,4,6-Trichlorophenol	3,030
		Pentachlorophenol	2,500
		2,4-Dimethylphenol	1,400
		Benzo(a)pyrene	1,300
		Benzo(k)fluoranthene	1,300
		Chrysene	1,300
		Fluoranthene	1,100
		Naphthalene	800
		Pyrene	950
		4-Nitrodiphenylamine	5,000
		n-Nitrosodiphenylamine	1,900
		Bis(2-chloroethyl)ether	2,900
		Bis(2-cloroisopropyl)ether	2,900
		Bis(2-ethylhexyl)phthalate	5,000
		Di-n-Butylphthalate	5,000
		3,3'-Dichlorobenzidene	8,500
		Hexachlorocyclopentadiene	10,000

1.2.2.5 Site S

Site S is located southwest of Site O. Allegedly, the property is, or was, owned by the Village of Sauget and the Resource Recovery Group. In the mid-1960s, solvent recovery began on site under Clayton Chemical, which is now owned by the Resource Recovery Group (RRG). The waste solvents were steam-stripped resulting in still bottoms that were allegedly disposed of in a shallow, on-site excavation that is now designated Site S. Historical aerial photographs indicate that Site S was potentially a waste and/or drum disposal area. The northern portion of the site is grassed and its southern portion is covered with gravel and fenced.

Soil samples collected from Site S are reported to contain VOCs, SVOCs, PCBs and metals at concentrations up to:

VOCs (ppb)	
Ethylbenzene	450,000
4-Methyl-2-Pentanone	93,000
Toluene	990,000
1,1,1-Trichloroethane	12,000
Xylenes	620,000

SVOCs (ppb)	
Naphthalene	200,000
Bis(2-ethylhexyl)phthalate	20,000,000
Butyl Benzyl Phthalate	490,000
Di-n-Butylphthalate	1,500,000
Di-n-Octylphthalate	310,000

PCBs (ppb)	
Aroclor 1248	85,000
Aroclor 1254	69,000
Aroclor 1260	41,000

Metals (ppm)	
Copper	139
Lead	0.392
Mercury	3.5
Zinc	327

Additional information regarding environmental investigation at the Sites may be found in the Site Sampling Plan.

1.3 PROJECT OBJECTIVES AND SCOPE

The purpose of this RI/FS is to gather sufficient information to quantify potential risks to ecological and human health receptors based on the presence of chemical residuals in environmental media. This QAPP reflects the objectives of the terrestrial portion of the ERA only, which is to determine if chemicals detected in soil will affect the terrestrial ecosystem.

Vegetation (e.g., grasses) will be collected to characterize if contaminant uptake is occurring in plants, and if so, the potential adverse effects to the prairie vole (*Microtus ochrogaster*) that consumes these plants. Because earthworms (*Lumbricus sp.*) ingest soil, they will be collected to characterize potential adverse impacts to a vermivore, the short-tailed shrew (*Blarina brevicauda*). In order to correlate with contaminant concentrations in soil, samples of biota will be collected from the same locations where soil/waste characterization samples are collected. Samples of biota will be analyzed for chemicals that may bioaccumulate including semi-volatile organic compounds (SVOCs), pesticides, herbicides, PCBs, dioxins/furans, and Target Analyte List (TAL) metals.

Due to sample size limitations for earthworms, sample analyses hierarchy will be performed, if necessary. Earthworm samples will be analyzed for PCBs first, followed by all other parameters including SVOCs, dioxins, herbicides, pesticides, lipids, and metals if sufficient sample mass can be collected.

1.4 SAMPLING PLAN DESIGN AND RATIONALE

The sampling plan design and rationale for sample locations for the ERA is described in Section 4 and in the separate Ecological Assessment Field Sampling Plan.

1.5 ANALYTICAL PARAMETERS, RATIONALE, MEDIA, AND FREQUENCY

Chemical data for all biota will be used in the Ecological Risk Assessment. Sample matrices, analytical parameters, and sample collection frequencies are shown in Tables 2 through 7. Project-required reporting limits (RL) for biota samples were developed through the USEPA Data Quality Objective (DQO) process (Section 3) and are based on data searches from the scientific literature for background concentrations and bioaccumulation information. Where this information was not available, method and practical limits of quantitation from laboratory instrumentation in tissues were used to form the basis for the RL. In cases where the laboratory reporting limit does not meet the ecological risk-based criterion, a footnote appears in the tables explaining the approach to report below the RLs down to the laboratory method detection limit (MDL).

The laboratories will report their MDLs as shown in the laboratories' QA Plans, included as Attachments A and B. The laboratory reporting limits will be supported by a low-level standard in their calibration curves (organic compounds) for all compounds for which they cannot achieve the project RLs. For those compounds that the laboratory reports between the RLs and the MDLs, the results will be flagged as estimated ("J"). This approach will generate the lowest level reporting using the laboratory protocols and USEPA methods described.

To meet the needs of this program, the project team will work together frequently to ensure that the resulting project RLs are as low as technically feasible and that sample analytical results will achieve RLs within the limits of the selected analytical methods. The usability of such data with higher RLs will be evaluated during the risk assessment activities. For purposes of performing risk assessment studies, levels of non-detected constituents will be assumed to be present at concentrations equal to one-half of the sample-specific reporting limit in media where at least one sample has a detected constituent[1].

Sample types and frequency are found in Section 4 of the QAPP and the separate FSP.

1.6 DATA QUALITY OBJECTIVES

The DQO Process is a series of planning steps designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended application. The DQO process presented below is consistent with USEPA guidance (USEPA, 1994b).

Step 1: State the problem.

This step involves a description of the problem(s) and specifications of available resources and relevant deadlines for the study.

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[1] For example, if a constituent is not detected in any sample, the concentration of that constituent will be assumed to be zero.

1. *Identify the members of the planning team.* The planning team is listed in Section 2 and Figure 3 of the QAPP. Planning has benefited from input from Charles R. Harman (AMEC), Steven Smith (Solutia), and Mike McAteer (USEPA RPM) as well as other technical personnel in these organizations and in supporting consulting firms.
2. *Identify the primary decision-maker.* The primary decision-maker is Steven Smith at Solutia and Mike McAteer at USEPA.
3. *Develop a concise description of the problem.* Chemical contamination has been detected in Sites O, P, Q, R, and S which may pose adverse ecological risks to the terrestrial biota inhabiting the sites or adjacent areas.
4. *Specify available resources and relevant deadlines for the study.* The PRP committee, Solutia, and AMEC will provide the resources necessary to meet the stated objectives. The project schedule is provided in Section 1.7. The ecological work will consist of a site reconnaissance survey, terrestrial biota sampling, and an ERA report, at dates to be determined.

Step 2: Identify the decision.

This step involves a statement of the decision that will use environmental data and the actions that could results from this decision.

1. *Identify the principal study decision* - Do chemical contaminants in soils, vegetation, and macroinvertebrates pose an unacceptable environmental risk to ecological receptors as represented by Assessment Endpoints?
2. *Define alternative actions that could results from resolution of the principal study question.* Information on ecological risk might be used to determine if any remedial activities are needed, plan remedial activities for environmental media within and adjacent to Sites O, P, Q, R and S, and/or determine the potential risks associated with remediation.
3. *Combine the principle study question and the alternative actions into a decision statement.* Decide if remedial activities are needed to reduce unacceptable risks to ecological receptors. Identify which risks need to be addressed. Decide if remediation would result in net environmental benefits to ecological receptors.

4. *Organize multiple decisions.* Decisions are organized as follows: How will unacceptable risks be determined? Are there unacceptable risks? What receptors and media contribute to risks? What remedial actions will reduce these risks to acceptable levels? What are the risks posed by these remedial steps? Will remediation result in net environmental benefits?

Step 3: Identify the inputs to the decision.

This step involves listing environmental variables or characteristics that will be measured and other information needed to resolve the decision statement.

1. *Identify the information that will be required to resolve the decision statement.* To resolve the decision statement, measurement of constituents in soil, vegetation, and macroinvertebrates and community evaluation of fauna and flora using multiple lines of evidence are necessary.
2. *Determine the sources for each item of the information identified.* Chemical measurements in biota (i.e., vegetation, earthworms) will be performed using standard USEPA methods (SW846) as described in Section 7 of this QAPP. Biota samples will be analyzed for those chemicals that bioaccumulate by standard USEPA methods as described subsequently in the QAPP. Sample media, analytical parameters, and frequencies of sample collection and measurements are shown in Tables 1 through 7.
3. *Identify the information necessary to establish the action level.* A discrete action level is inappropriate for evaluating ecological risk; therefore, the multiple lines of evidence approach will be used. In accordance with this method, the risk assessor needs the following information: 1) the confidence in each measure of risk (measurement endpoint), 2) the response in the measure, based on chemical/toxicological results, and 3) the concordance among measures (variability). This approach is described in the ERA Workplan. As an initial screening level of risk, and to help establish project-specific reporting limit requirements, risk-based concentrations (RBCs) of contaminants to be measured in biota were established to help set the DQOs for the ERA (Table 8).
4. *Confirm that appropriate measurement methods exist to provide the necessary data.* Analyses of biota samples will be performed in strict accordance with USEPA methods

(SW846, latest edition) with appropriate modifications for tissue extractions and cleanup procedures (Section 7). Chemical measurement methods must be able to meet sensitivity requirements for the ERA; therefore, RLs for biota samples were developed through the USEPA DQO process through searches of background concentrations and bioaccumulation information available in the scientific literature. Where this information was not available (e.g., herbicides in biota), method and practical limits of quantitation, from tissue analysis in the laboratories formed the basis for the RL. In cases where the laboratory reporting limit does not meet the ecological risk-based criteria, a footnote appears in the tables (Tables 2 through 7) explaining the approach to report below the RLs down to the laboratory detection limit to obtain the necessary data.

Step 4: Define the boundaries of the study.

A detailed description of the spatial and temporal boundaries of the problem, characteristics that define the populations of interest, and any practical considerations of interest are described in this step.

1. *Specify the characteristics that define the population of interest.* Local populations of ecological resources include those individuals that inhabit or feed from the areas around Sites O, P, Q, R, and S.
2. *Define the spatial boundary of the decision statement*
   a. *Define the geographic area to which the decision statement applies* - For the ecological assessment, the geographic boundaries include Sites O, P, Q, R, and S. The proposed sample locations for biota collection are the same locations from which surface soil and waste characterization samples will be gathered..
   b. *When appropriate, divide the population into strata that have relatively homogenous characteristics.* The local populations will be considered one at Sites O, P, Q, R, and S.
3. *Define the temporal boundary of the decision statement.*
   a. *Determine the timeframe to which the decision statement applies.* It will be assumed that samples collected during 2001 represent current conditions.

b. *Determine when to collect the data.* Data collection activities will be performed during Summer 2001.

4. *Define the scale of decision making.* The assessment will be based on historical data as well as the information gathered during 2001. The decision will be made at spatial scales appropriate to the selected Assessment Endpoints.
5. *Identify practical constraints on data collection* - Collection efforts will be influenced by weather conditions (especially rain), availability of biota for collection for sample analysis, as well as by the suitability of habitats to support biota. Matrix effects on the accuracy of the chemical measurements for some tissue analyses may pose a practical constraint on the data usability for ecological risk calculations. Such effects will be minimized by using cleanup techniques in the laboratory during sample preparation (Section 7).

Step 5: Develop a decision rule.

This step involves defining the parameter(s) of interest, specifying the action level and integrating previous DQO outputs into a single statement that describes a logical basis for choosing among alternative actions.

1. *Specify the statistical parameter that characterizes the population of interest.* A number of statistical methods will be utilized to evaluate risk; however, chemical concentration data will be expressed as arithmetic means, median, and 95th upper confidence intervals on the mean (or maximum values if the data set is too small) for risk assessment calculations.
2. *Specify the action level for the study.* There is no single action level for establishing ecological risk, it is dependent on integrating multiple lines of evidence that include toxicity benchmarks, community analyses, and toxicity tests. Final remediation goals will be determined when the remedy is selected. Remediation goals will establish acceptable exposure levels that are protective of ecological receptors.
3. *Develop a decision rule.* The multiple lines of evidence approach will be used which considers the confidence in each measure of risk (measurement endpoint), the response in that measure, and the concordance amongst measures. This approach is described in the ERA Workplan.

Step 6: Specify the tolerable limits on decision errors.

This step involves determining tolerable decision error rates based on a consideration of the consequences of making a decision error.

1. *Determine the possible range of the parameter of interest.* The measurement methods defined can accommodate a wide range of chemical concentrations for each analyte of interest. The project-specific RLs were developed using this DQO process, to meet the data input requirements for the ecological risk assessment through data usefulness. The historical range of chemicals of interest will be reviewed and discussed in the ERA report.
2. *Identify the decision errors and choose the main hypothesis.*
   a. *Define both types of decision errors and establish the true state of nature for each decision error.* The two types of decision error are false negatives and false positives.
   b. *Specify and evaluate the potential consequences of each decision error.* False negatives could result in unresolved risks; the second can result in expenditures for unnecessary remediation and additional ecological harm.
   c. *Establish which decision error has more severe consequences near the action level.* False negatives are considered to have more severe consequences to the environment.
3. *Specify the range of possible values of the parameters of interest where the consequences of decision errors are relatively minor (gray region).* The "gray region" is represented by equivocal results from multiple lines of evidence approach and response that are considered "small" (e.g., toxicity differences on the order of 30%).
4. *Assign probability values to points above and below the action level that reflect the tolerable probability for the occurrence of decision errors.* Not applicable.

Step 7: Optimize the plan.

1. *Review the DQO outputs and existing environmental data.* There is no existing biota data for use in the ERA. Historical data for other media will be reviewed and incorporated, if acceptable, as part of the ERA report. The DQO outputs for this project, based upon this DQO process, are described in detail in Section 3.

2. *Develop the general data collection design.* The sampling plan design and rationale for sample locations in support of the ecological assessment is summarized in Section 4 and the separate FSP and explained in greater detail in the Ecological Risk Assessment Workplan and the RI/FS Support Sampling Plan.

   The highest level of data quality is defined for data generated in support of risk assessment. Analyses will be performed in strict accordance with the USEPA methods defined in Section 7 of the QAPP. Chemical data used in the ERA will be validated in accordance with USEPA data validation guidance, and the QA/QC requirements described in the QAPP. Specific DQOs for QA/QC to support the ERA chemical measurements have been defined for the QA/QC parameters of accuracy, precision, sensitivity, representativeness, completeness, and comparability and are described in Section 3.

1.7 PROJECT SCHEDULE

The project schedule for sampling and analysis in support of the Sauget Area 2 Sites ERA is summarized below and depicted in Figure 4.

- late August 2001 — Site Reconnaissance
- October - November 2001 — Aquatic/Terrestrial Sampling Activities
- October 2001 - January 2002 — Laboratory Analysis
- November - December 2001 — Data Validation
- April 2002 — Report Submission

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The project organization and responsibilities for the Sites environmental activities is defined in this section. A Project Organization Chart is presented in Figure 3 and includes the individuals discussed below.

2.1 USEPA PROJECT MANAGER

The USEPA Region 5 Project Manager, Mike McAteer, has the overall responsibility for the environmental activities in support of the Sauget Area 2 Sites RI/FS activities.

2.2 USEPA FIELD SERVICE STATION

The USEPA Field Service Station may assist the USEPA RPM in project support through the technical review of documents, plans, and data, as necessary.

2.3 ILLINOIS ENVIRONMENTAL PROTECTION AGENCY PROJECT MANAGER

The IEPA Project Manager, Candy Morin, has the overall responsibility of ensuring that the project meets the IEPA objectives and quality standards.

2.4 SITE PROGRAM MANAGER

The Site Program Manager and Chairman of the Sauget Area 2 Sites Group, Steven Smith of Solutia, Inc. has the overall responsibility for ensuring that the project meets USEPA objectives and quality standards. In addition, he is responsible for technical quality control and project implementation and oversight. The Site Program Manager will ensure that technical, financial, and scheduling objectives are achieved successfully. The Site Program Manager will report directly to the USEPA Region 5 Project Manager and will provide the major point of contact and control regarding the project. The Site Program Manager responsibilities include the following:

- Define project objectives and develop detailed workplans and schedule with the project team;
- Establish project policy and procedures to address the specific needs of the entire project as well as those of each task;

- Acquire and apply technical and corporate resources as needed, to ensure performance with budget and schedule constraints.
- Orient all field leaders and project team staff concerning the project's special considerations;
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned requirements;
- Approve all deliverables prior to their submission to the USEPA Region 5 Project Manager
- Ultimate responsibility for the preparation and quality of interim and final reports; and
- Represent the project team at meetings and public hearings.

2.5 Ecological Project Manager and Field Leader for Ecological Risk Assessment

The Site Manager will be supported by the Ecological Project Manager and Field Leader for the Ecological Risk Assessment. AMEC Earth and Environmental, Inc. (AMEC) of Somerset, NJ will perform the sampling and analysis activities to support the Ecological Risk Assessment evaluation at the Sites. Charles R. Harman, PWS will provide the high-level technical direction for the ERA. The Ecological Project Manager is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision in support of the ERA activities. The Ecological Project Manager will report directly to the Site Program Manager. Specific Ecological Project Manager/Field Leader responsibilities include the following:

- Provision of day-to-day coordination with the Site Program Manager on technical issues concerning the sampling and analysis of biota for the ERA;
- Development and implementation of the ERA Workplan and this QAPP, including the specific field sampling plan activities described in Section 4;
- Coordination and management of field staff for the collection of biota samples and documentation of field observations important for the ERA evaluation;

- Implementation of QAPP procedures for the collection and analysis of samples from biota;
- Adherence to work schedules provided by the Site Program Manager;
- Identification of problems at the field team level, discussion of resolutions and implementation of corrective actions, as necessary; and
- Prepare, review, and approve the ERA for the Sauget Area 2 Sites investigation including coordination and oversight of technical efforts of subcontractors assisting the ERA team.

2.6 ECOLOGICAL QA CHEMISTS

Quality Assurance (QA) oversight for the ERA sampling and analysis activities described in this QAPP/FSP will be provided by Ms. Elizabeth Wessling and Ravichandran Mahalingam, Ph.D., both of AMEC. Responsibilities include:

- Preparation of the QAPP/FSP in support of the ERA;
- Development of project DQOs to support the ERA activities;
- Coordination with the analytical laboratory and field teams, as necessary, to ensure proper implementation of QAPP/FSP procedures;
- Coordination with the data validation team, as necessary, to determine data usability for ecological risk evaluation;
- Technical assistance to the Ecological Project Manager and the Site Program Manager, as necessary for chemistry and QA-related issues.

2.7 TECHNICAL STAFF FOR THE ECOLOGICAL RISK ASSESSMENT ACTIVITIES

The technical staff for the ERA will be assembled from the AMEC staff and will be utilized to gather and analyze data and to prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the ERA work. The technical staff includes field observation and field sample collection staff, ecological risk assessors, QA professionals, and regulatory experts.

2.8 LABORATORY QUALITY ASSURANCE OFFICERS AND PROJECT MANAGERS

Responsibilities of the analytical laboratories, STL - Savannah and Triangle Laboratories are described in the attached QAPPs, provided as Attachments A and B, respectively. STL-Savannah will be used for analysis of SVOCs, PCBs, pesticides, herbicides, and metals. The Project Manager is Michelle Owens at 912-354-7858, the Laboratory Manager is C. Henry Beauchamp and the QA Manager is Kirstin McCracken.

Dioxin analyses will be completed by Triangle Laboratories. The Project Manager is Helen Smpardos at 919-544-5729 and the QA Manager is Greg Johnson.

2.9 DATA VALIDATION

Data validation will be performed by AMEC Earth & Environmental, Inc. supervised by Ms. Elizabeth Wessling of the Denver, CO office and Ravichandran Mahalingam, Ph.D of the Atlanta, GA office.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The DQO Process is a series of planning steps designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended application. The DQO process presented is consistent with USEPA guidance (USEPA, 1994b). DQOs are quantitative and qualitative statements derived from outputs of each step of the DQO process that:

- Clarify the study objective;
- Define the most appropriate type of data to collect; and
- Determine the most appropriate conditions from which to collect the data.

The DQO process is developed through a multi-step process that was described in detail in Section 1.

The overall QA objective for this project is to develop and implement procedures for field sampling, laboratory analysis, chain-of-custody, and reporting for biota samples that will provide technically valid results for use in the Ecological Risk Assessment. This section provides specific project DQOs and intended data usages that were developed through the DQO process. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventive maintenance, and corrective action are described in other sections of this QAPP.

3.1 LEVEL OF QUALITY CONTROL EFFORT

The following specific quality control parameters will be collected, prepared, and analyzed to evaluate the quality of the data generated to support the ERA. The DQOs for Precision and Accuracy are summarized in Tables 9 to 14. Sample types, criteria and corrective actions are summarized in Table 15.

Field blanks, laboratory method blanks, field duplicates, matrix spikes and matrix spike duplicates (MS/MSDs), laboratory control samples (LCS), surrogates, laboratory calibration QC, and tissue standard reference materials (SRM) will be analyzed to assess the quality of the data resulting from field sampling and analysis of environmental media.

3.1.1 Field/Trip Blanks

Field blanks are equipment rinsate blanks for the same analytical parameters as for the samples (SVOCs, pesticides, herbicides, dioxins, PCBs, and metals) and are intended to demonstrate field decontamination efficiency. The field blank will consist of laboratory provided, distilled interference-free water poured over the appropriate sampling equipment then preserved with the appropriate preservative (Table 16). Field blanks will be collected from trowels/shovels used to collect earthworms and from shears/scissors used to collect vegetation. Field blanks are collected at a rate of one per matrix per decontamination event.

The trip blank is necessary only if aqueous samples are collected for VOC analysis. Therefore, trip blanks are not warranted for the terrestrial ERA sampling.

3.1.2 Field Duplicates

Field duplicates provide a measure of reproducibility (precision) for the sampling procedures and the representativeness of the samples. Two co-located samples from a single location are obtained and prepared and analyzed by the laboratory. Each sample is labeled with a unique sample number and both are submitted to the laboratory for separate analysis. Field duplicates are collected at the frequency of one per ten samples collected per medium. Frequency and acceptance criteria are defined in Tables 9 through 14. For this project, two duplicates will be collected for vegetation and earthworms.

3.1.3 Method Blanks

Method blank samples are generated within the laboratory and are used to assess contaminated resulting from laboratory procedures. Results from the method blanks provide an estimate of the variability with batches of the blank response and indicative of bias introduced by preparation and analytical procedures. Method blank analysis must be performed for each extraction or digestion batch for each analytical method at a minimum frequency of one method blank per 20 field samples. Frequency and acceptance criteria for method blank acceptance are listed in Tables 9 through 14.

3.1.4 Laboratory or Matrix Duplicates

Duplicate samples are two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. In the laboratory, duplicate samples or matrix duplicates (MD) are analyzed to check for sampling and analytical reproducibility as a measure of precision and representativeness. The duplicate sample is a separate aliquot that the laboratory prepares and analyzed identically to the original sample. The relative percent difference between the duplicate results is a measure of precision and representativeness. Criteria for laboratory matrix duplicates for all compounds of interest in biota are listed in Tables 9 through 14. Note that for organic analysis (Table 9), the matrix duplicate precision requirements are equivalent to those indicated for the field duplicate precision.

3.1.5 Matrix Spikes and Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicates (MSD) provide information about the effect of the sample matrix (environmental medium) on the digestion and measurement methodologies. One MS/MSD pair must be generated for every 20 or fewer biota samples for both organic and inorganic analysis. Criteria for acceptance are based upon percent recoveries of the MS or MSD and are defined in Tables 9 through 14 and are based upon those acceptance limits provided by the USEPA Contract Laboratory Program (CLP). However, as required by SW-846, each laboratory must routinely update the accuracy limits based upon their experience with real-world samples. Therefore, the limits achieved by the laboratories for accuracy may be different than those indicated in these tables.

The relative percent difference of the MS/MSD results also gives a measure of the precision and representativeness of the organic data (Tables 9 through 14).

3.1.6. Laboratory Control Sample/Standard Reference Material

A laboratory control sample (LCS) and/or standard reference material (SRM) will be prepared and analyzed with each batch of field biota samples or at a minimum frequency of one LCS/SRM per 20 biota samples. The LCS/SRM will contain the compounds of interest for organics and inorganics in an appropriate tissue matrix as available from a reliable, verifiable source (certified vendor, NIST). The results of the LCS/SRM must meet vendor's limits for acceptance and measures the method accuracy (Table 13).

For organics, standard reference materials will be obtained for tissues (biota), as available, for the compounds of interest. Vendor-generated 95% confidence limits will be the acceptance criteria for the SRMs. Frequency for SRMs is one per 20 samples per laboratory sample batch.

3.1.7 Surrogate Spikes

A surrogate spike contains pure substances not usually found in nature, with properties that mimic the compounds of interest. This spike is added to all organic samples prior to extraction to assess the method accuracy in the sample matrix. Criteria for surrogate spike recoveries are listed in Tables 9 through 14, based upon those acceptance limits given in the USEPA CLP. However, as required by SW-846, each laboratory must routinely update the accuracy limits based upon their experience with real-works samples. Therefore, the limits achieved by the laboratories for accuracy may be different than those indicated in these tables.

3.1.8 Laboratory Calibration Check Samples

A variety of QC samples are analyzed for separate analytical methods to assess the accuracy of the analysis on a day-to-day basis. These QC checks, include but are not limited to the following: criteria for initial calibration, continuing calibration, baseline drift and contamination which are performed per method requirements by the laboratory. The details of these QC checks are available in the methods referenced in Section 7 and the laboratory-specific SOPs for analysis. A summary is presented in Table 15.

3.2 PRECISION

Precision is a measure of the degree to which two or more measurements are in agreement. Field and laboratory precision QC requirements are listed in Tables 9 through 14. Field and laboratory precision will be assessed through the calculation of relative percent differences (RPD) of field duplicate results, MSD results, and MD results. The equations for the calculation of precision criteria are listed in Section 12.

3.2.1 Field Precision Objectives

Field precision will be assessed through the collection and measurement of field duplicates, as described in Section 3.1.2.

3.2.2 Laboratory Precision Objectives

Laboratory precision will be assessed through the preparation and analysis of MSD samples for organic compounds and MD samples for metals and organic compounds results, as described in Section 3.1.2.

3.3 ACCURACY

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. Accuracy will be assessed through the evaluation of recoveries of spiked compounds of interest into biota samples, as well as the evaluation of SRM for tissues and through the evaluation of field and laboratory blanks. Accuracy criteria for MS/MSD samples, LCS, and blanks are provided in Tables 9 through 14. The limits in these tables are those which have been established through the USEPA CLP; however, as required by SW-846, each laboratory must routinely update the accuracy limits based upon their experience with real-works samples. Therefore, the limits achieved by the laboratories for accuracy may be different than those indicated in these tables. The equations used for accuracy are listed in Section 12.

3.3.1 Field Accuracy Objectives

Field accuracy will be assessed through strict adherence to sample handling protocols and laboratory preservation and analysis holding times to maintain the integrity of the biota sample.

3.3.2 Laboratory Accuracy Objectives

Laboratory accuracy will be assessed through method blank analysis (Section 3.1.3), MS/MSD (Section 3.1.5), SRM/LCS (Section 3.1.6), surrogate compound spikes (Section 3.1.7), laboratory calibration checks (Section 3.1.8), and the determination of percent recoveries of the QC samples. Accuracy control limits are given in Table 9 through 14 and also in the applicable SOPs as referenced in Section 7. Note that all chemicals of concern included in Tables 2 through 7 must be included in method spiking solutions for the LCS and MS/MSD samples.

3.4 Sensitivity - Reporting Limit Requirements

The sensitivity or reporting limit requirements for this project were defined to meet ERA requirements. The compounds of concern, sampling media, and ecological project-required reporting limits for the level of detection are presented in Tables 2 through 7.

These reporting limits will be achieved in tissue samples through following the procedures, as specified in Section 7. Note that the achievable reporting limits in tissue samples may be affected by matrix interferences. Sample cleanups, such as gel permeation chromatography (GPC) and silica gel, may be performed by the laboratory to minimize matrix effects and to obtain project reporting limits.

The rationale for the project reporting limits and discussion of approach to report to the MDLs when necessary to achieve risk-based levels of detection were discussed in Section 1.5.

3.5 COMPLETENESS

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected under normal conditions. The equation for completeness is presented in Section 12.

3.5.1 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all field measurements taken for the project. The field completeness objective for this project is greater than 90 percent.

3.5.2 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all laboratory measurements taken for the project. The laboratory completeness objective for this project is greater than 90 percent.

3.6 REPRESENTATIVENESS

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process conditions, or an environmental conditions within a defined spatial and/or temporal boundary. Representativeness is dependent upon the proper design of the sampling program. The field sampling rationale has been developed to collect representative biota samples to assess the potential for ecological impacts at Sauget Area 2 and is discussed in the Ecological Risk Assessment Workplan and the Ecological Risk Assessment Field Sampling Plan (Section 4).

3.6.1 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied if the procedures described in the Ecological Risk Assessment Workplan and the Ecological Risk Assessment Field Sampling Plan are followed. The media of concern are biota which include vegetation and earthworms. One measure of representativeness includes the precision of field duplicate measurements (Section 3.1.2).

3.6.2 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the analytical procedures defined in Section 7, maintaining proper preservation and meeting sample holding times to maintain sample integrity, performing appropriate homogenization and aliquoting procedures to ensure representative samples for analysis, and analyzing and assessing field and laboratory duplicate samples (Section 3.1.4).

3.7 COMPARABILITY

Comparability is an expression of confidence which one data set can be compared to another. Comparability is dependent upon the proper design of the field sampling and analytical measurement program.

3.7.1 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied through adherence to the Ecological Assessment Field Sampling Plan (Section 4).

3.7.2 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented, as required by the QAPP. Comparability is also dependent on consistent QA objectives. As such, comparability of biota data will be achieved by following sampling procedures for biota collection as described in Section 4, by using standard USEPA tissue analysis methods (with modification for tissue extraction, as described in Section 7), and by evaluating the data validity and usability using standard USEPA procedures and QA/QC criteria described in Section 9.

4.0 ECOLOGICAL ASSESSMENT FIELD SAMPLING PLAN

This section is the Field Sampling Plan (FSP) for the terrestrial portion of the Ecological Risk Assessment. It describes the conceptual approach for the sampling event, locations for collection of biota samples, collection procedures, numbers of samples to be collected, labeling and chain-of-custody requirements, and sample container and preservation and holding time requirements.

4.1 STUDY AREA

The ecological assessment focuses on Sites O, P, Q, R, and S, as described in Section 1.2.

4.2 FIELD SAMPLING RATIONAL AND SAMPLING LOCATIONS

The terrestrial portion of the Ecological Assessment Field Sampling Plan consists of two separate field events: a reconnaissance survey and the sampling event.

4.2.1 Reconnaissance Survey Objectives

A reconnaissance survey will be conducted in early May 2001 to refine the field sampling activities proposed in the following section. The observations made during the survey will be used to provide a verbal and photographic description of the sites, finalize sampling locations, procedures, and the number of biota samples that can be realistically collected during the sampling program. The objectives and justification for the Survey activities are described below.

- Photodocument Sites O, P, Q, R, and S.
- Locate previous soil/waste characterization sample locations from the RI to co-locate biota samples from same area.
- Finalize representative receptor species for use as assessment endpoints.
- Conduct a qualitative fauna/flora survey and habitat and cover type assessments. Direct and indirect (e.g., calls, scat, tracks) observations will be documented.
- Determine the dominant vegetation species and relative abundance for subsequent sampling.

- Determine the most appropriate sampling techniques.

The results of the survey will be summarized in a concise technical memorandum for submission to the USEPA. The technical memo will summarize the chemical results of screening sample analysis, present the observations made during the recon survey, and confirm the position to collect terrestrial and aquatic samples during the field work. Modifications to the field sampling program as a result of the reconnaissance survey will be documented in an amendment to this QAPP and the Field Sampling Plan. . USEPA approval will be required for the workplan prior to the field sampling effort.

4.2.2 Sampling Program

During the sampling program, biota samples will be collected for laboratory analysis of target compounds. The objectives of the sampling program are as follows:

- Collect vegetation and earthworms in Sites O, P, Q, R, and S for chemical analyses of tissues. Concentrations of target analytes in biotic tissue will be used in dietary exposure models for the selected receptor species for extrapolation to assessment endpoints. Information on tissue analyses will be used to evaluate potential effects on the terrestrial foodchain.
- The objective of vegetation sampling is to determine concentrations of target analytes for use in exposure models for the representative prairie vole; the objective of the earthworm sampling is to determine concentrations of contaminants in earthworms due to the earthworm's ingestion of contaminated soil for use in exposure models for the short-tailed shrews. Both rodents are prey for the red fox (*Vulpes vulpes*), the terrestrial receptor. Body burdens in the short-tailed shrews will be estimated using earthworm body burdens and exposure models such as those presented by USEPA (1999).
- Biota tissue samples will be stored in dry ice prior to and for shipment to the laboratory. At the laboratory, biota samples will be stored frozen prior to chemical analysis, as described in Table 17. Frozen storage of tissue samples can be maintained for a maximum of one year, consistent with USEPA guidance on solid

and tissue sample preservation (40 CFR 136.3). The method-specified holding times for extraction and analyses begin when samples are thawed for preparation and analysis. Preservation and holding times are listed in Table 16.

4.2.3 Sample Locations

Four soil and waste characterization samples are proposed for collection in each of Sites O, P, R, and S during other RI/FS field activities. Because of the relative size of Site Q compared to the other areas, an additional four soil and waste characterization samples will be collected there. The sampling of biota will be co-located with surface soil samples to determine if chemical contamination in soils poses adverse ecological risks to terrestrial biota inhabiting or feeding from the sites. The surface soil samples in all Sites will be located in the worst case locations based on the results of a reconnaissance using electromagnetic induction (EMI) and soil gas survey equipment to be conducted by URS. However, if the worst case locations chosen by URS do not have any habitat for the selected ecological receptors (other than in Level A), the field crew has the option to collect another surface soil sample at the nearest location providing suitable habitat. Soil sampled for the ecological risk assessment should be sieved to remove large pieces of gravel and vegetation as these pieces do not contribute directly to either earthworm, plant or wildlife incidental ingestion of soil. The sample will be analyzed for the same parameters as the URS sample, including total organic carbon, and used for the location of the earthworm and plant samples.

4.3 VEGETATION SAMPLING

The goal of vegetation sampling is to collect a sufficient amount of vegetative matter from the dominant species for chemical analysis at each of the 24 soil sample locations.

4.3.1 Vegetation Sample Collection

Subsequent to classification of the herbivorous vegetation (e.g., grasses), the dominant species will be determined and sufficient sample (approximately 25 grams per fraction will be required for SVOCs, pesticides, herbicides, PCBs, dioxins, and metals) will be collected to make a composite of 175 to 200 grams for laboratory analysis.

4.3.2 Vegetation Sample Analytes, Containers, and Shipment Requirements

Terrestrial plants will be collected for chemical analysis of tissues to estimate exposure to rodents, such as the prairie vole, that may feed on the vegetation. To minimize variability associated with differential uptake by plant species, an effort will be made to analyze the same or similar plant species at all locations. The selection of species will be guided by observations made during the reconnaissance survey.

Different portions of the plant can be used as a food source; however, for the purposes of this RI/FS, only aboveground portion (stems/leaves/seeds) will be collected for chemical analysis. Each sample will be a composite of enough vegetation to comprise sufficient sample for analysis. Vegetation will be collected using decontaminated stainless steel scissors/shears. The composite will be washed with distilled water to remove soil from the vegetation. While it is readily accepted that dry deposition can account for a significant portion of foliar contaminant levels, the objective of the sampling is to assess the potential for contaminant uptake from the soils to the leaf structures. Dry deposition and particulate resuspension in many ways cannot be directly attributed to site-related activities for which the PRPs are responsible. Therefore, the leaves will be washed with distilled water to remove that confounding factor. The sample will then be placed in a sealable plastic bag and placed on dry ice for shipment to the laboratory for analysis of SVOCs, pesticides, herbicides, PCBs, dioxins, percent moisture, and metals. Each sample will be assigned a unique number and will also be labeled with the data of collection, time, and initials of the collector. Sample analyses, preservation, containers and holding time requirements are provided in Table 16.

4.4 EARTHWORM SAMPLING

The goal of macroinvertebrate sampling is to obtain sufficient earthworms biomass for tissue analysis of chemicals at each of 24 soil sample locations.

4.4.1 Earthworm Sample Collection

Earthworms will be collected by digging through soil using a field decontaminated, stainless steel trowel or shovel, as necessary. The laboratory requires approximately 25 grams of sample per sample fraction (i.e., SVOCs, herbicides, pesticides, PCBs, dioxins, metals, lipids) for a total sample of 175 to 200 grams. Additional sample material will be necessary for matrix QC, including the MS/MSD and matrix duplicate. The following scheme will be used to achieve sufficient earthworm sample size within a reasonable time period for the sampling program.

- The sampling team will initially dig a hole to a depth of approximately six inches at the location of the soil sample. An additional effort will be made to cut around the edges "as quickly and deeply as possible" excavating a hole approximately 25 to 40 cm across and 10 to 30 cm deep (10-16 in and 4-12 in, respectively) (James 1996). The root mat will be carefully disaggregated and searched for worms Depending on the amount of earthworms encountered, additional soil will be removed from the hole until enough worms have been collected.
- After collection is completed, samples will be washed and rinsed with distilled water, placed in a sealable plastic bag, and stored on dry ice for shipment to the laboratory.

4.4.2 Macroinvertebrate Sample Analytes, Containers, and Shipment Requirements

Earthworms will be collected for chemical analysis of tissues to estimate exposure to rodents, such as the short-tailed shrew, that consume earthworms. Each sample will be a composite of enough earthworms to allow for chemical analysis of all parameters, if possible. The composite will be washed to remove soil from the outside of the earthworms[2] then placed in a sealable plastic bag and placed on dry ice for shipment to the laboratory for analysis of SVOCs, pesticides, herbicides, PCBs, dioxins, lipids, and metals. Each sample will be assigned a unique number and will also be labeled with the data of collection, time, and initials of the collector. Sample analyses, preservation, containers and holding time requirements are provided in Table 16.

[2] Earthworms will not be depurated prior to chemical analysis to simulate actual conditions.

5.0 SAMPLE CUSTODY

Chain-of-custody (COC) procedures for biota collection will follow custody protocols as described in USEPA guidance (USEPA, 1985). This custody protocol is compliant with USEPA Region 5 requirements for sample custody and is divided into three parts: field-specific sample collection, laboratory custody, and final evidence files.

A sample or evidence file is under custody if:

- the item is in your possession;
- the item is in your view, after being in your possession;
- the item is in your possession and you have placed it in a secured location;
- the item is in a designated secure area.

5.1 Field Chain of Custody Procedures

The sample packaging and shipment procedures summarized below will ensure that samples will arrive under proper chain-of-custody. Specific sample numbering protocols are described in Section 4.

5.1.1 Field Procedures

(a) The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. The number of persons handling the samples should be kept to a minimum to preserve sample integrity.

(b) All bottles will be identified with unique sample numbers and locations on secure bottle labels that will include sample identification numbers, location, date of collection, time of collection, and type of analysis required.

(c) Sample labels will be marked with waterproof ink.

(d) Samples will be accompanied with a properly completed COC form (Figure 5) that will include sample numbers and locations. Further field custody documentation and transfer procedures are described in Sections 5.1.2 and 5.1.3.

5.1.2 Field Logbooks/Documentation

Field logbooks will provide the means of recording data collection activities. As such, entries will be described in as much detail as possible so that persons subsequently entering the site may reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook with be identified by the project-specific document number. The title page of each logbook will contain the following:

- person to whom the logbook is assigned.
- logbook number
- project name
- project start date, and
- project end date.

Entries made at the beginning of each entry will include the date, start time, weather, names of all sampling team members present and their affiliation, level of personal protection being used, and the signature of the person making the entry. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will also be recorded in the field logbook.

Measurements made, photographs taken, and samples collected will be recorded. All entries must be made in black ink. No erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark and initialed and dated by the person correcting it. Whenever a sample is collected or a measurement is made, a detailed description of the station location, including compass and distance measurements, shall be recorded. The number of the photographs taken of the location will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

The procedures and equipment used to collect samples will be noted along with the time of sampling, sample description, depth at which the sample was collected (as applicable), and amount and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples will receive a unique sampling number and will also be recorded in the field logbook.

5.1.3 Transfer of Custody and Shipment Procedures

The sample packaging and shipment procedures summarized below will ensure that samples will arrive with proper COC.

(a) Samples are accompanied by a properly completed COC form that will include sample numbers and locations. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and record the time on the COC. The COC documents the transfer of sample custody from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

(b) Samples will be properly packaged for shipment, including ice to preserve all samples at $\leq 4^{\circ}C$ and dispatched to the appropriate laboratory for analysis, with a separate, signed COC form enclosed in each shipping container. Shipping containers will be secured with strapping tape and custody seals will be affixed prior to shipment to the laboratory.

(c) All shipments will be accompanied by the COC record identifying the contents. The original record will accompany the shipment and copies of the COC will be retained by the field personnel for documentation. It is recommended that a copy of the COC be faxed to the laboratory on the date of collection.

(d) If the samples are sent by common carrier, a bill of lading (e.g., airbill) should be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody form as long as the custody forms remain sealed inside the sample cooler and the custody seals remain intact.

5.2 LABORATORY CHAIN OF CUSTODY PROCEDURES

Laboratory custody procedures for sample receiving and log-on, sample storage, tracking during sample preparation and analysis, and storage of data are described in the laboratories' QAPPs and SOPs (see Attachments A and B).

5.3 FINAL EVIDENCE FILES CUSTODY PROCEDURES.

The final evidence files for the data supporting the ERA will be maintained by the Site Program Manager at Solutia. The content of the evidence file will include, at a minimum, all relevant records, reports, correspondence, logs, field logbooks, pictures, subcontractor's reports including data validation reports, assessment reports, progress reports, and chain-of-custody records/forms. The evidence file will be under custody of the Site Program Manager in a locked, secured area.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

All instruments used to perform chemical measurements must be properly calibrated prior to, and during, use to ensure acceptable and valid results. This section describes the procedures necessary for maintaining the accuracy of all the instrumentation used in the field test and the laboratory analysis. The accuracy and traceability of all calibration standards used must be properly documented. The procedures described herein are to be used in conjunction with the specific instrument manufacturer's instruction, applicable analytical methodology requirements, and specific laboratory/field procedures for instrument operation.

6.1 FIELD INSTRUMENTS/EQUIPMENT

Field measurements are not planned for the ERA sampling activities described herein.

6.2 LABORATORY INSTRUMENTS

The methodologies selected for use in this investigation specify the types and frequency of calibrations. For all analytical procedures, the lowest calibration standard specified must be at or below the project required reporting limit for the specific media being tested to ensure accurate reporting limit determination. The specific methods to be used are provided in the laboratories' QA Manuals that detail specific instrumentation and calibration procedures.

Accessory analytical equipment such as refrigerators, balances and ovens required for the storage and preparation of samples must be calibrated using manufacturer's instructions with the following guidelines:

- Calibrations of equipment must be checked daily and these records kept in a logbook or calibration-specific log.
- The laboratory must clearly document the acceptance criteria for all such equipment (e.g., refrigerator temperature must be $4 \pm 2^{\circ}C$) and corrective actions must be taken for any out-of-control situation as described in the laboratory's quality assurance plan or manual.

- The equipment must not be used after corrective action until it has been recalibrated or verified through the successful analysis of a check standard.
- Calibrations of other miscellaneous analytical equipment (e.g., automatic pipettes) must be performed according to the manufacturer's recommendations.

Implementation of the laboratory calibration will be the responsibility of the Laboratory Director and the analysts performing the procedures.

7.0 ANALYTICAL PROCEDURES

This section describes a brief overview of the analytical methodologies to be used during the terrestrial component of the Sauget Area 2 Ecological Risk Assessment.

7.1 FIELD ANALYTICAL PROCEDURES

Field measurements will not be conducted as part of this assessment.

7.2 LABORATORY ANALYTICAL PROCEDURES

Laboratory analyses for SVOCs, pesticides, herbicides, PCBs, and metals will be performed by Severn-Trent Laboratories (STL) - Savannah. Dioxins and dibenzofurans will be analyzed by Triangle Laboratories. Details on laboratory analyses and QA procedures can be found in the laboratories' QA Plans (Attachments A and B, respectively).

7.2.1 Biota Methods

Analysis of vegetation and earthworms will be conducted off-site by STL-Savannah and Triangle Laboratories, in accordance with the USEPA methods summarized in Table 17. In addition, all soil samples will analyzed for total organic carbon, all plant and earthworm samples will be analyzed for percent moisture, and earthworm samples will be analyzed for lipids. The corresponding analytical parameters and project-required reporting limits are listed in Tables 2 through 7. These values are compared with risk-based levels for food items presented in Table 8. Those reporting levels presented in Tables 2 through 7 that exceed the calculated risk-based levels are identified with an asterisk. In cases where the laboratory reporting limit does not meet the ecological risk-based criterion, a footnote appears in the tables explaining the approach to report below the RLs down to the laboratory method detection limit (MDL). USEPA Region 5 Ecological Screening Levels (USEPA, 1998) were not used because there were no levels presented for whole body chemical concentrations in prey species. Additional guidance is provided as follows:

- Parameters will be analyzed according to analytical procedures set forth in the USEPA Test Methods for Evaluation Solid Waste, Physical/Chemical Methods, SW 846, 3rd Edition, Final Update, December 1996.
- Sample preparation for biota samples (vegetation, earthworms) prior to solvent extraction or digestion will include homogenization of each sample using a tissuemizer or blender at the laboratory. This procedure will ensure a uniform sample aliquot for analysis.
- Samples that have significant matrix interferences may require specialized cleanup procedures an/or re-analysis in order to eliminate interferences and to permit analysis to proceed with a reporting limit at or closer to the project-required reporting limit. Any matrix interference that results in elevated reporting limits without positive results for target analytes must be reported by the laboratory. Cleanup protocols will be anticipated for the biota sample analyses. Gel-permeation chromatography (GPC USEPA SW-846, Method 3640A) may be used on solvent extracts for organic compounds prior to analysis to remove high molecular weight fatty acids and lipids. Additional cleanup procedures may be required and, if necessary, will be drawn from the procedures given in SW846.

The laboratory will maintain current SOPs for extraction, cleanup, and analysis of biota material and must have on file, current MDL studies, as shown in the QA Plans (Attachments A and B) to demonstrate their ability to meet the project required reporting limits. The MDLs must be performed by the laboratory on an annual basis to ensure the ongoing ability to perform the methods as specified. The MDLs will be performed in accordance with USEPA guidance described in 40 CFR 136, 1986, Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11".

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 FIELD MEASUREMENTS

Onsite field measurements will not be conducted for the terrestrial portion of the ERA.

8.2 LABORATORY ANALYSIS

Laboratory QC checks include the analyses of initial and continuing calibration checks, blanks, spiked samples (MS/MSD, LCS and/or SRM) analysis, cleanup check samples, surrogates (organics only), laboratory duplicate samples (matrix duplicates),and retention time window determination for applicable organic methods. A brief description of each of these check samples is given below. Criteria that the laboratory must meet for these are based on the specific analytical methods used and are summarized in Tables 9 through 14. Laboratory QC will be checked against the analytical methods and data usability criteria during the data generation and review process.

8.2.1 Calibration Criteria

Calibration checks will be performed according to the method-specific requirements as summarized below. The specifics for the calibrations are detailed in the individual analytical methods.

8.2.2.1 Organic Analysis

- Multilevel initial calibrations (usually 5-level) will be performed to establish the instrument's response to the targets of interest across a range of concentrations (calibration curves). The lowest level calibration standard must be at or below the project-required reporting limit.
- Calibration verification will be performed at least once every 12 hours of gas chromatograph/mass spectrometer (GC/MS) analysis. For GC analyses, verification will occur every ten samples of GC instrument analysis to ensure continued accurate quantitation.

- GC/MS instrument tuning systems will be performed every 12 hours using the method-appropriate tuning standard and acceptable criteria.

8.2.1.2 Inorganic Analysis

- Multilevel calibration curves generated by analyses of individual or mixed standards.
- Initial calibration verification at the beginning of each run and continuing calibration verification at a minimum of one every ten samples to verify ongoing instrument performance.
- Inductively coupled plasma (ICP) interference check standards after initial calibration and after sample analysis (within eight hours) to verify inter-element and background corrections.

8.2.2 Blanks

Method blanks are generated by the laboratory as they are processing field samples. Method blanks are analyte-free matrices that are processed using all of the reagents and procedures that are used on the field samples to evaluate the presence of contamination during sample preparation and analysis. Method blanks will be analyzed at a minimum of one per 20 field samples per matrix per preparation batch. Contamination found in the method blank and similarly in the field samples may be an indication of cross-contamination and may not be indicative of environmental contamination. Additional method blanks, such as cleanup method blanks, may be generated to independently verify the cleanup technique, if used. Criteria for method blank acceptance is method-specific and shown on Tables 9 through 14.

Analytical blanks are required for inorganic analyses during initial and continuing calibration verification and are analyzed at the beginning, during, and end of the analytical sequence to assess contamination and instrument drift. The initial calibration blank (ICB) is run after the initial calibration verification (CV) and prior to sample analysis. The continuing calibration blank (CCB) is analyzed every ten samples following the ICB throughout the analytical run and at the end of the sequence. These blanks are prepared by acidifying the reagent water to the same concentrations of acids found in the samples and standards. Acceptance criteria for analytical blanks are the same as for method blanks (Tables 9 through 14).

8.2.3 Matrix Spikes and Matrix Spike Duplicates

Matrix spike samples are prepared by spiking known concentrations of target analytes into an aliquot of field sample. The MS is processed in exactly the same manner as all other field samples. The percent recovery of the target spike compound is an indication of the ability of the analytical method, and of the laboratory, to accurately quantitate the target analyte in the spiked sample. The MS recovery may aid the analyst in determining whether a matrix effect or interference exists in the analysis of the unspiked sample. For organic analyses, the MS recovery does not necessarily reflect the ability to accurately determine the target analyte or analytes of similar chemical nature in other field samples. MS target compounds and criteria are method-specific and are summarized in Tables 9 through 14.

8.2.4 Surrogate Spikes

All samples, including field and QC samples that are analyzed for organic components will have surrogates[3] added to the samples during preparation procedures. The surrogates used are method-specific and are similar in chemical nature to the targets of interest; however, they are not normally found in environmental samples. The surrogate compound recoveries assist the analyst and data user in determining the accuracy of the measurements for the target compounds of interest (Tables 9 through 13).

8.2.5 Laboratory Control Samples and Standard Reference Material

Laboratory control samples (LCS) are prepared by spiking known concentrations of target analytes into analyte-free matrices (blank matrices). Standard reference material (SRM) contains the analytes of interest in a matrix of interest and are purchased from a standards vendor. LCS and SRM are prepared and analyzed concurrently with field samples. Target recovery from the LCS/SRM is a measure of the ability of the preparation and analysis methods to accurately quantitate target analytes in the absence of matrix effects or interferences. LCS will be analyzed at a minimum of one per 20 field samples per matrix per preparation batch. LCS criteria are analyte and method-specific and are summarized in Tables 9 through 14. The

[3] In dioxin analyses, the term for surrogates is "sample fortification mixture".

SRM criteria are based on the manufacturer's accuracy limits. The laboratory will obtain appropriate SRM for analysis with biota samples. If an SRM is used as a measure of method accuracy for target analytes in biota, then an LCS not required.

8.2.6 Cleanup Check Samples

Whenever a cleanup technique (e.g., GPC, alumina column cleanup, etc.) is employed to eliminate interferences which may prevent accurate determination of targets of interest at the project-required reporting limit, the cleanup procedures must be verified through the analysis of check standards. A standard containing some or all of the target analytes must be processed through the cleanup procedure and analyzed. The recovery of the target analytes in this check will indicate if cleanup procedures were effective in elimination of interferences without undo removal of targets of interest.

8.2.7 Laboratory Duplicates

A laboratory matrix duplicate is a separate sample aliquot taken from the same sample container as a field sample and is prepared and analyzed independently. Comparison of all positive results between the sample and matrix duplicate, through determination of the RPD, provides a measure of the analytical precision and accuracy of the quantitation. A sample/MD pair will be prepared and analyzed at a frequency of one per 20 samples per matrix per analytical batch. RPD acceptable criteria for the sample/MD are analyte and method-specific and are summarized in Tables 9 through 14. Note that for organic analyses, the precision criteria for field duplicates are equivalent to those for the sample/MD precision.

8.2.8 Retention Time Window Determination

For organic analyses, the target analyte retention time window will be determined based on the procedure specified in the analytical method. Positive identification of an analyte will be made when its retention time falls within the window established during calibration.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

All data generated by the laboratories shall be reduced, reviewed, and validated prior to use in the ERA using the following procedures.

9.1 DATA REDUCTION

9.1.1 Field Data Reduction Procedures

Field measurements are not part of the field activities associated with the terrestrial portion of the ERA. Field activities include observations and sample collection only.

9.1.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures will be performed according to the following general protocols and laboratory-specific protocols as described in the laboratories' QA Plans (Attachments A and B). All raw analytical data will be recorded and documented using laboratory standard procedures. Laboratory data will include, at a minimum, the unique sample identification number, analytical method used, name of analyst, the data of analysis, matrix samples, reagent and standard concentrations, instrument settings, final results, units, and sample-specific reporting limits. Periodic review of laboratory notebooks (logbooks) and data reports shall be performed by the Lab QA Manager as described in the laboratory QAPP.

For this project, analytical results for all biota samples will be calculated and reported on a wet-weight basis. QC data (e.g., laboratory duplicates, surrogates, MS/MSDs) will be compared to the acceptance criteria defined in Section 3 and 7 of this QAPP. Laboratory case narratives will be prepared which will include information concerning data that are outside acceptance limits and any other anomalous conditions encountered during sample analysis. After the laboratory submits the data package to the Site Program Manager, the data are considered approved by the laboratory and read for data validation.

9.2 DATA VALIDATION

Formal data validation, using standard USEPA protocols for evaluating the technical and regulatory validity of environmental data shall be performed for laboratory-generated chemical data. For field activities, informal data review of observations and documentation will be performed.

9.2.1 Procedures Used to Validate Field Data

The procedures to evaluate field information for the ERA include checking for transcription errors and review of field logbooks which will be performed by the AMEC field team leader.

9.2.2 Procedures Used to Validate Laboratory Data

Procedures to validate laboratory data will be derived from the USEPA guidance (1994a, 1994c). These protocols will be modified to include the criteria listed in Sections 3 and 8 of this QAPP.

The validation includes a review of all technical holding times, instrument performance check sample results, initial and continuing calibration results, and all batch and matrix QC including field blanks, field duplicates, MS/MSD, matrix duplicates, surrogate recoveries, method blanks, laboratory control samples, standard reference material results and the identification and quantitation of specific compounds of interest. One hundred percent of the analytical data will be validated.

Additionally, MDL studies for all chemicals of concern in tissues will be performed by the laboratory. The MDLs will support the project reporting limit requirements and will have been performed within one year of the Sauget ERA sample collection. The laboratory shall follow the MDL procedures as outlined in the Federal Register[4] and associated laboratory QAPP SOPs.

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[4] MDL procedures to be followed are outlined in the Federal Register 49(209), pp 198-199, October 26, 1984.

The laboratory's current MDLs for the analytes and matrices of interest are provided in Attachments A and B.

In addition to the precision, accuracy, and sensitivity criteria as defined in Section 3 and 8, the overall completeness of the data package will also be evaluated by the Data Validator. Completeness checks will be administered on all data to determine whether deliverables specified in the following section are present. The reviewer will determine whether all required items are present and request copies of missing deliverables, using resubmittal request documentation via facsimile or e-mail. Such documentation will be included in the data validation reports.

9.3 DATA REPORTING

9.3.1 Field Data Reporting

No field measurements are planned for the terrestrial portion of the ERA.

9.3.2 Laboratory Data Reporting

The laboratory will provide at least two hard copies of each laboratory data report, an original and a copy for data validation, to the Site Program Manager. Electronic deliverables will be required for the project database. Specific formats for electronic deliverables shall be determined by the Site Program Manager, the Ecological Risk Assessors and Data Validators at AMEC, and the analytical laboratory prior to the start of the program.

The laboratory data reports shall consists of the following, at a minimum:

1. Case Narrative

    - Date of issuance
    - Laboratory analysis performed
    - Any deviations from intended analytical strategy.
    - Laboratory batch number

- Numbers of samples and respective matrices
- QC procedures utilized and also references to the acceptance criteria
- Laboratory report contents
- Project name and number
- Conditions of samples "as received"
- Discussion of whether or not samples holding times were met
- Discussion of technical problems or other observations which may have created analytical difficulties
- Discussion of any laboratory QC checks which failed to meet project criteria
- Signature of the Laboratory QA Manager and/or Laboratory Director or designee

2. Chemistry Data Package

- Summary page indicating dates of analyses for samples and laboratory QC checks
- Cross referencing of laboratory sample identification numbers to project sample identification numbers
- Description of laboratory data qualifiers used
- Sample preparation and analyses dates and methods used for samples
- Sample results in wet weight with units clearly labeled
- Sample-specific reporting limits
- Raw data for sample results and laboratory QC samples
- Results of (dated) initial and continuing calibration checks, and GC/MS tuning
- MS/MSD recoveries and relative percent difference (RPD), MD results and sample/MD RPD, laboratory control samples/standard reference recoveries, method blank results
- Calibration check compounds, system performance check results, surrogate recoveries
- Chromatograms/spectra or other raw data of sample results and QC checks
- Example result calculations

The data package submitted will be a "CLP-like" data package consisting of all the information presented in a CLP data package, including CLP-like reporting forms to facilitate data validation. Tentatively identified compounds (TICs) will not be reported.

9.4 DATA RECONCILIATION WITH ECOLOGICAL RISK ASSESSMENT REQUIREMENTS FOR USABILITY

The goal of this project is to produce an ecological risk assessment. As such, the data generated must meet the risk assessor's needs as defined in Section 3, which are:

1) Collect data that are representative of site conditions and comparable with prior data
2) Produce data that meets the project reporting limit requirements
3) Produce data of the highest quality possible to accurately and precisely characterize the Site ecological conditions.

The Data Validation team will apply the standard data validation qualifiers to data to indicate the level of uncertainty in the associated result. In general, data that are left unqualified, data qualified "U" (not detected), data qualified as "J" (estimated concentration), and data qualified as "UJ" (not detected at an estimated MDL) are considered valid and usable for project objectives. Data that are qualified "R" (rejected) due to severe exceedances of QC requirements will be considered invalid and unusable in the ERA.

The goal of this QAPP/FSP program is to generate valid, usable data for the ERA; however, some data may be lost due to sampling location logistics, field or laboratory errors, or matrix effects that may cause the rejection of some results. The overall completeness of valid data collection, as defined in Section 3 is 90%. The Data Validation team will assess the completeness of the overall data generation against the project goal of producing 90% of the planned data as valid and useable results for the ERA. If this goal is not met, data gaps may exist that may compromise the ERA.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with the procedures established in this QAPP/FSP and the Ecological Risk Assessment Workplan. Field and laboratory audits will include two independent parts - internal and external audits.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

10.1.1 Internal Field Audit Responsibilities, Frequency, and Procedures

Internal audits of field activities (sampling and field observations) will be conducted by the Ecological Project Manager/Field Team Leaders to verify that established procedures are being followed. Internal field audits should be conducted at least once at the sampling commencement and potentially during the course of sampling activities, should problems arise. Follow-up audits may be conducted to correct deficiencies and to verify that QA procedures are maintained throughout the project.

Internal field audits will include examination of field sampling records, field observation records, sample collection handling and packaging in compliance with the established procedures, defined in Sections 4 and 5.

10.1.2 External Field Audit Responsibilities, Frequency, and Procedures

An external audit may be conducted as required by the appropriate USEPA Region 5 QA staff or designee. External field audits may be conducted any time during field operation and may or may not be announced at the discretion of USEPA Region 5.

External field audits, if performed, will be conducted according to the field activity information presented in Sections 4 and 5 and field activities described in the ERA Workplan. The external field audit process may include, but is not limited to, sampling equipment decontamination procedures, sample bottle preparation procedures, sampling procedures, examination of field sampling and safety plans, sample vessel cleanliness and QA procedures, procedures for verification of field duplicates, sample preservation and preparation for shipment, and chain-of-custody procedures.

10.2 LABORATORY PERFORMANCE AND SYSTEM AUDITS

10.2.1 Internal Laboratory Audit Responsibilities, Frequency, and Procedures

The internal laboratory audit will be conducted by the Laboratory QA Officer. Internal systems audits will be performed on an annual basis; internal performance audits on a quarterly basis in accordance with laboratory QA procedures.

The internal system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. The auditor should ensure that all SOPs and MDLs are current and appropriate for the matrices and analyses being conducted for the project. The laboratory internal auditor will follow procedures described in the laboratory QA Plan for internal system audits.

The performance audits may involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The laboratory QA Officer will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance. The laboratory auditor will follow procedures for the performance audits as described in the laboratory QA Plan. Data package review, as discussed below, may also be performed.

10.2.2. External Laboratory Audit Responsibilities, Frequency, and Procedures

An external laboratory audit may be conducted as required by appropriate QA staff of USEPA Region 5 or designee. AMEC does not plan to conduct an external performance evaluation of the analytical laboratories as they have been pre-qualified to perform chemical analysis for this project based on prior performance on other projects for Solutia and by maintaining appropriate QA/QC procedures, as evidenced by their QA Manuals, SOPs, and MDLs. Additionally, 100% of the chemical data generated for the ERA will be validated under USEPA CLP protocols. The validation will determine QA/QC issues that may affect the data. The USEPA and the project team reserve the option to perform an external audit of the laboratories if deemed necessary to the success of the project.

External laboratory audits may be conducted any time during the analytical operations and may or may not be announced at the discretion of USEPA Region 5 or designee.

External audits may include the following: review of laboratory analytical procedures, laboratory onsite visits and results of performance evaluation samples submitted to the laboratory for analysis. Failure of any or all audit procedures chosen can lead to laboratory disqualification and the requirement than another suitable laboratory be chosen.

An external on-site review can consist of sample receipt procedures, custody and sample security and log-in procedures, sample storage procedures, review of instrument calibration records, instrument logs and statistics (number and type), review of QA procedures, log books, sample preparation procedures, analytical SOP review, MDL review, instrument reviews, personnel interviews, review of glassware preparation procedures, and corrective action protocols.

While conducting an external laboratory audit, one or more data packages from sample lots recently analyzed by the laboratory will be reviewed. This review will most likely include, but is not limited to:

- comparison of resulting data to the SOP or method, including deviation.
- verification of initial and continuing calibrations within control limits
- verification of surrogate recoveries and instrument timing results, where applicable.
- review of extended quantitation reports for comparison of library spectra to instrument spectra, where applicable.
- recoveries on laboratory control samples and/or SRM analyses.
- review of run logs with run times, ensuring proper order of runs.
- review of spike recoveries/QC sample data

- review of suspected manually integrated GC data and its cause, where applicable.
- review of GC peak retention times and resolution for compounds as compared to reference spectra, where applicable.
- assurance that samples are run within holding times.

11.0 PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

Field measurements are not scheduled for the terrestrial portion of the ERA.

11.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE

A routine preventative maintenance program is conducted by the laboratory to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees regularly perform routine scheduled maintenance and repair of (or coordinate vendor maintenance) all instruments. All laboratory instruments are maintained in accordance with manufacturer specifications. The details of the preventative maintenance procedures are included in the laboratories' QA Manuals (Attachments A and B) and are not reiterated herein. In general terms, the preventative maintenance program includes the following steps:

- An inventory of replacement and spare parts for instruments that are maintained.
- Maintenance logbooks for each instrument to be kept along with information on routine and non-routine procedures. The logbook records must include the instrument number, date of maintenance activity, and the type of activity performed.
- Training laboratory staff in the instrument maintenance requirements. Preventive maintenance schedules and activities will be outlined in the laboratory SOPs and will be adhered to.

The following sections describe the general preventative maintenance procedures for major pieces of analytical equipment. The specific laboratory QA Manuals should be consulted for specific procedures for each laboratory.

11.2.1 Inductively Couple Plasma Spectroscopy

The ICP spectrometer should be maintained under service contract with the manufacturer. Routine preventive maintenance should include:

- Inspect pump tubing and replacement, as necessary.

- Inspect nebulizer for even spray and clean, as necessary.
- Inspect torch for plasma height and shape and clean, as necessary.
- Inspect photomultiplier sensitivity and replace as necessary.

11.2.2 GC/MS Instruments

The GC and GC/MS systems will be maintained on a service contract or undergo in-house maintenance to provide routine preventive maintenance. Spare parts for the GC and GC/MS systems should include filaments, electron multiplier, source parts, o-rings, ferrules, septa, injection port liners, and columns. Routine preventive maintenance for the systems should include:

- Inspection of data systems (disk drives, tape readers, etc.) and servicing, as necessary.
- Change oil and traps on mechanical and turbo pumps.
- Service MS source through cleaning, replacement of filaments and other source parts and necessary.
- Replace injection port septa and liners, as necessary.
- Clipping front end of GC column or replacement, as necessary.

11.2.3 Thermometers

Thermometers for refrigerators and ovens are calibrated yearly against National Institute of Standards and Technology (NIST) certified thermometers. The laboratory QA manager will be responsible for the safekeeping of the NIST thermometers and for the documentation asserting the accuracy of their measurements.

11.2.4 Analytical Balances

Virtually every analytical procedure requires the use of side-loading and/or top loading balances. Many of these requirements involve standards preparation which are crucial to accurate determination. Balances should be maintained on a service contract. A calibration status label is affixed to each balance after calibration during servicing.

12.0 SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The purpose of this section is to indicate the methods by which samples will be in compliance with the DQOs described earlier in this QAPP. A combination of statistical procedures and qualitative evaluations will be used to check the data quality. These procedures will be used by the laboratory in generating the data and by the Data Validator in validating data for use in the ERA.

Results for QC samples, including field and laboratory blanks, spikes, and duplicates as previously described in Sections 3, 6, and 8 will be evaluated using the equations described below to determine data usability and validity. In addition, the data will be reviewed for indications of interferences to results from sample matrices, cross contamination (field or laboratory), and sample preservation and storage anomalies. The following procedures refer to laboratory-generated chemical data in biota samples.

12.1 PRECISION ASSESSMENT

The relative percent difference (RPD) is a measure of variability between the MS and MSD for organics or between the sample and matrix duplicate in the case of inorganics and field duplicate pair will be calculated compare to precision and representativeness DQOs. The RPD of duplicate measurements is calculated according to the following formula:

$$RPD = \frac{(\text{Result in Sample 1} - \text{Result in Sample 2})}{\frac{(\text{Result in Sample 1} - \text{Result in Sample 2})}{2}} \times 100$$

where:

Sample 1 = Initial sample or spike sample result

Sample 2 = duplicate sample or duplicate spike sample result

12.2 ACCURACY ASSESSMENT

Accuracy, as a measure of bias, will be evaluated based on the percent recoveries of the matrix spike sample (organic and inorganics), matrix spike duplicate sample (organics), surrogates (organics), internal standards (organics), LCS and/or SRM (organics and inorganics), initial and continuing calibration check samples (organics and inorganics). These QC results will be compared to the project DQOs for accuracy.

The increase in analyte concentration observed in the spiked sample, due to the addition of a known analyte quantity, is compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Percent recoveries for spike samples and QC are determined using the following equation.

$$\%R = \frac{\text{(Result in spike sample - Result in original/unspiked sample)}}{\text{known amount of spike added}} \times 100$$

Percent recoveries for LCS/SRM are determined using the following equation:

$$\%R = \frac{\text{Result for compound in LCS or SRM}}{\text{Verified amount of compound in LCS or SRM from vendor information}} \times 100$$

Additionally, field and laboratory blanks will be used to evaluate whether field or laboratory procedures represent a possible source of contamination in the biota samples. Unmonitored contamination results in reported false positives that are treated as true sample components when they are not. This type of error will adversely affect the accuracy of the reported results. Several types of blanks, including field blanks, method blanks, and instrument blanks, will be used in this project.

Specific DQOs for blanks have been defined for this program in Sections 3, 6, and 8. In general, the procedure for assessing blank samples for potential contamination is as follows:

- Tabulate blank compound results.
- Identify blank samples for which compounds are reported above the project-required reporting limits.
- If no compounds are detected above the reporting limits in any blanks, the associated data are reported unqualified and no blank actions are taken.
- If compounds are detected above the reporting limits in the blanks, the associated sample compounds will be qualified during data validation. This qualification may result in the negation of results at raised reporting limits due to blank actions.

Further details on blank actions are provided in the USEPA data validation protocols.

12.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid samples results to the total number of results planned for collection. Following completion of the sample, analysis, and data validation, the percent completeness will be calculated and compared to the project DQO of $\geq$90% using the following equation.

$$\%\ \text{Completeness} = \frac{\text{number of valid/usable results obtained}}{\text{number of valid/usable results planned}} \times 100$$

12.4 OVERALL ASSESSMENT OF ECOLOGICAL DATA

Data assessment will involve data validation and usability to determine if the data collected are of the appropriate quality, quantity and representativeness to support the ERA. The affect of lost data deemed unacceptable for use, for whatever reason, will be discussed and decision made on corrective action for potential data gaps. The QC results associated with each analytical parameter for each biota type will be compared to the objectives presented in Sections 3, 6, and 8. Only data generated in association with QC results meeting these objectives and the data validation criteria will be considered usable for the ERA.

Factors to be considered in the overall data assessment and based on the DQOs and data validation results will include, but are not limited to, the following:

- Samples obtained using methodologies and SOPs proposed in the QAPP.
- Proposed analyses performed according to the SOPs provided in the QAPP.
- Samples obtained from all proposed sampling locations.
- Elevated reporting limits due to matrix interferences or high concentrations of contaminants.
- Data validation conducted in accordance with USEPA CLP protocols, including project-specific QC objectives, as defined in the QAPP.
- Unusable ("R") and usable data based on the results of the data validation.
- Sufficient data in appropriate quality generated to support the ERA.
- Resolution of corrective actions.
- Remaining data gaps.

13.0 CORRECTIVE ACTIONS

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out of QC performance which can affect data quality and usability. Corrective actions may be required for two classes of problems - analytical and compliance/noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling and preparation, laboratory instrumental analysis, and data review.

For noncompliance problems, a formal corrective action will be implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Site Project Manager. A description of the problem and the corrective action implemented will be confirmed in writing via e-mail, facsimile, or technical memorandum.

Any nonconformance with the established quality control procedures in this QAPP will be identified and corrected. Corrective actions in the field will be implemented and documented in the field logbook.

13.1 FIELD SAMPLE COLLECTION

Technical staff and field project personnel will be responsible for reporting all suspected technical or QA nonconformance or suspected deficiencies of any field collection or observations activity by reporting the situation to the Ecological Project Manager/Field Leader. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the field personnel and a copy forwarded to the Site Program Manager.

The Ecological Project Manager/Field Leader will be responsible for ensuring that corrective action for nonconformance are initiated by:

- evaluating all reported nonconformance;
- controlling additional work on nonconforming items;

- determining disposition or action to be taken;
- maintaining a nonconformance logbook
- reviewing nonconformance reports and corrective actions taken;
- ensuring nonconformance reports are forwarded to the Site Program manager to be included in the final site documentation in project files.

If appropriate, the Site Program Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed. If a corrective action warrants a change in the program protocols, this change will be documented and signed by the AMEC Field Team Leader for the ERA and the USEPA RPM.

13.2 LABORATORY CORRECTIVE ACTIONS

The laboratories participating in this program are required to have written SOPs specifying corrective actions to be taken when an analytical error is discovered or the analytical system is determine to be out of control. The SOP requires documentation of the corrective action and notification by the analyst about the errors and corrective procedures. Additionally, corrective action procedures are included in the laboratories QA Plans.

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is dependent on the analysis and the event. Laboratory corrective action may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy;
- Blanks contain compounds of interest, as listed in Tables 1 through 6, above the project-reporting limits;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the laboratory QA department during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedures if filed with the QA department.

Corrective action may include:

- Reanalysis of samples, if holding times permits.
- Resampling and analysis.
- Evaluation and amendment of analytical procedures.
- Acceptance of data and acknowledgement of the level of uncertainty as documented in the laboratory data package case narrative.

If resampling is deemed necessary due to laboratory problems, the Site Program Manager must identify the necessary approach including cost recovery for the additional sampling effort.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The deliverables associated with the tasks identified in the ERA Workplan will contain QA sections in which data quality information collected during the task is summarized. The ERA Report will include the results of the data validation as documentation of data quality collected for assessing ecological risk.

The QA section of the ERA report will contain information generated during the project on the achievement of project-specific DQOs, uncertainties in the biota data used and their affect on the risk assessment, and a summary of corrective actions implemented, as necessary, as it may have affected the evaluation of ecological risk.

15.0 REFERENCES

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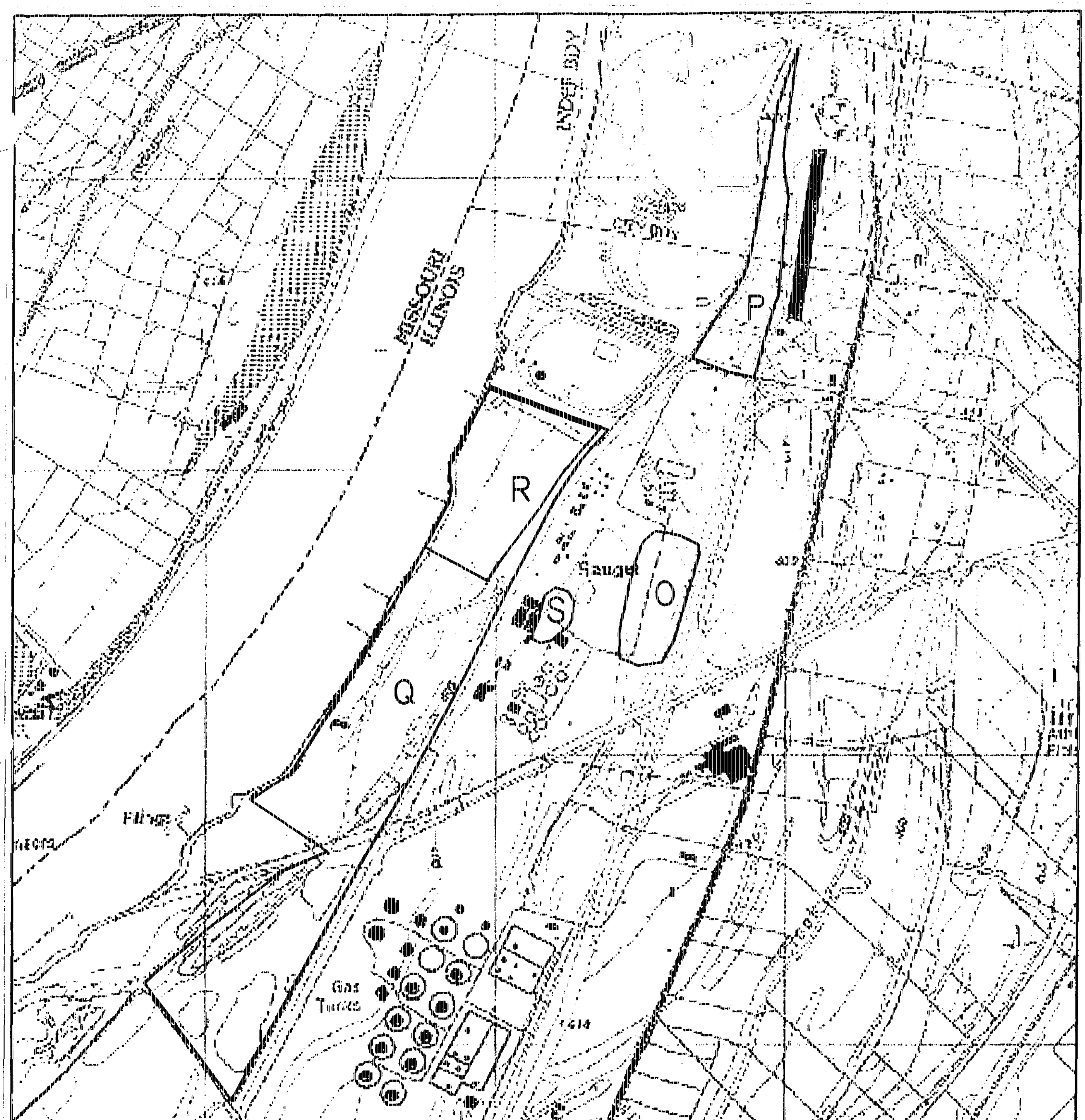
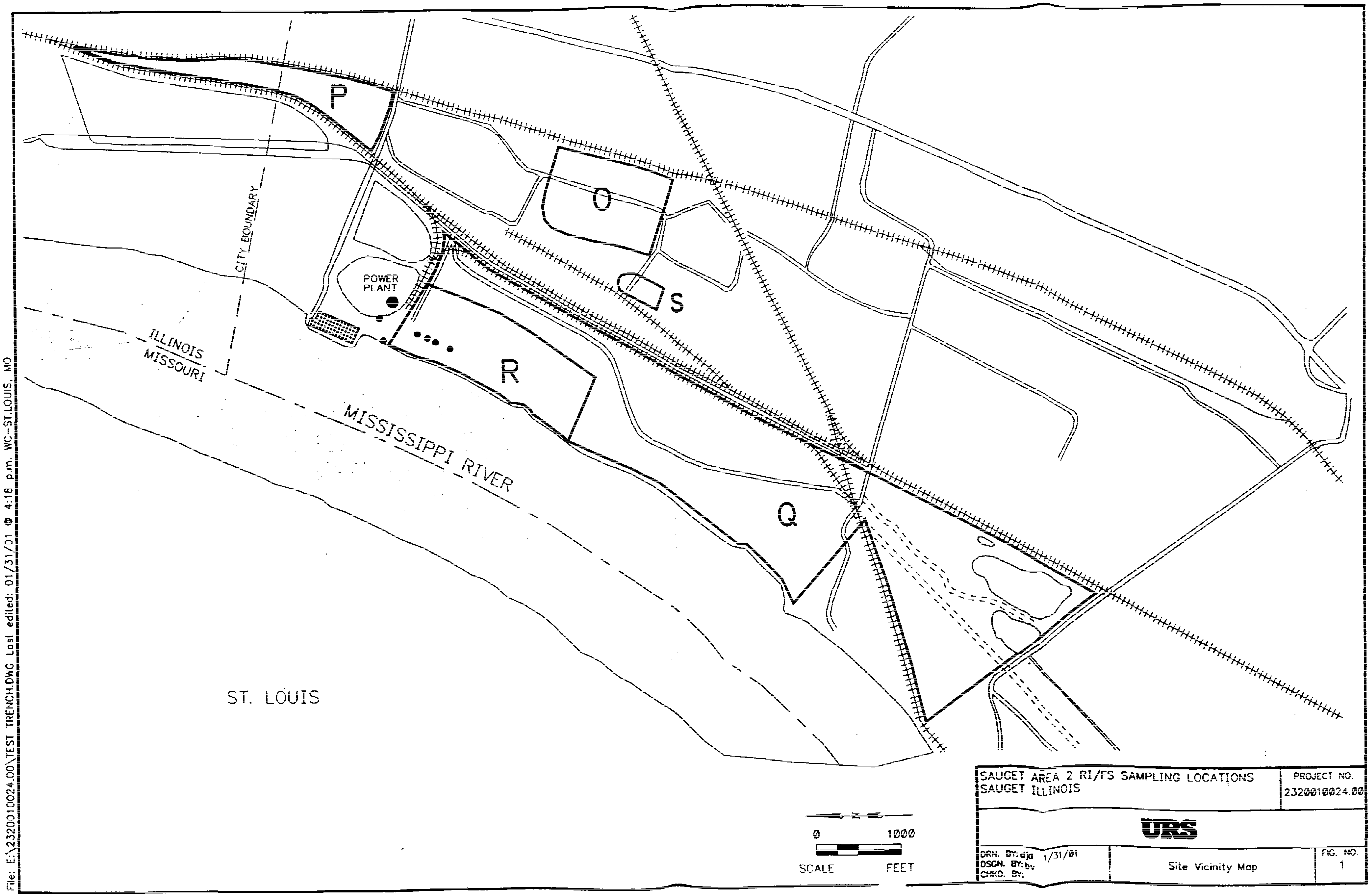


SOURCE: USGS QUADRANGLE (CAHOKIA, IL-MO), 1998
NOT TO SCALE

FIGURE 1

SITE LOCATION MAP

SAUGET AREA 2
SITES O, P Q, R, S
SAUGET AND CAHOKIA, ILLINOIS



SAUGET AREA 2 RI/FS SAMPLING LOCATIONS
SAUGET ILLINOIS

PROJECT NO. 2320010024.00

URS

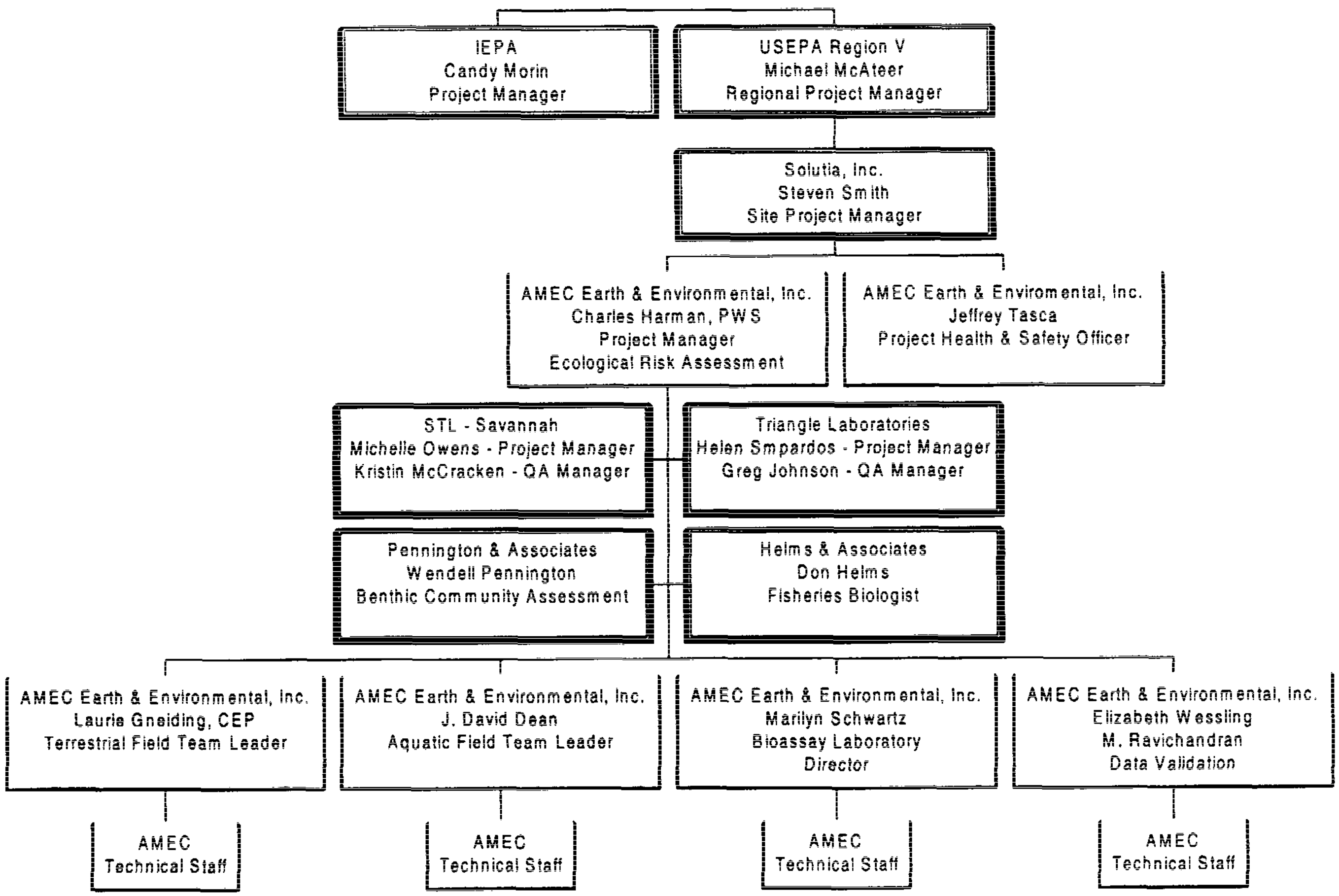
DRN. BY: djd 1/31/01
DSGN. BY: bv
CHKD. BY:

Site Vicinity Map

FIG. NO. 1

File: E:\2320010024.00\TEST TRENCH.DWG Last edited: 01/31/01 @ 4:18 p.m. WC-ST.LOUIS, MO

Figure 3
Project Team Organization Chart
Sauget Area 2 Sites
Ecological Risk Assessment

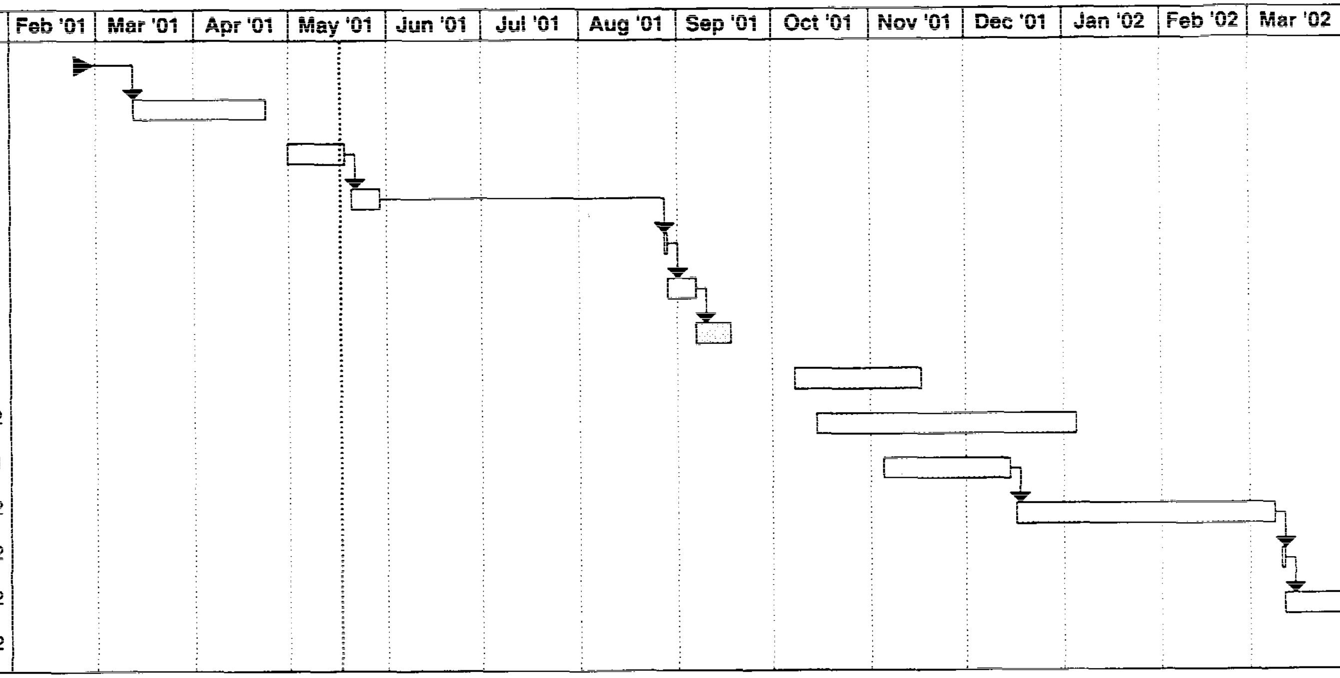


F 4

Sauget Area 2 Sites

Ecological Risk Assessment Project Schedule

ID	Task Name	Duratior	Start	Finish
1	Submit Plans to USEPA Region '	1d	02/22/01	02/22/01
2	USEPA Region V Review	30d	03/13/01	04/23/01
3	Response to USEPA comments	14d	05/01/01	05/18/01
4	Implementation/Mobilization	7d	05/21/01	05/29/01
5	Site Reconnaissance	1d	08/28/01	08/28/01
6	Modify Plans	7d	08/29/01	09/06/01
7	USEPA Workplan Approval	7d	09/07/01	09/17/01
8	Terrestrial/Aquatic Fieldwork	30d	10/08/01	11/16/01
9	Laboratory Analysis	60d	10/15/01	01/04/02
10	Data Validation	30d	11/05/01	12/14/01
11	Report Preparation	60d	12/17/01	03/08/02
12	Submission to Solutia	1d	03/11/02	03/11/02
13	Response to Solutia Comments	14d	03/12/02	03/29/02
14	Submission to USEPA Region V	1d	04/01/02	04/01/02



Severn Trent Laboratories
628 Route 10
Whippany NJ 07981
Tel: (973) 428-8181
Fax: (973) 428-5222

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CHAIN OF CUSTODY

No. 1304

FIELD BOOK: ______________ Pg _____ of _____

1 Client:

2 Project Name/no.:

3 Client Contact:

4 STL Contact:

5 TAT: 1wk, 2wk, 3wk, OTHER ________

6 Proj. Type: NJPDES, NPDES, ISRA, CLP, CERCLA, RCRA, UST, ACO, MOA, OTHER ________

7 Protocol: CLP, SW846, EPA 600 DW, OTHER ________

8 Reporting Type: NJ Reg Format, NJ Reduced Format, CLP, Level II, Level I (Data Sum), Other ________

14 Bill To

PO#

15 ANALYSIS REQUIRED

9 Client ID (10 CHAR)	10 Date	11 Time	12 Mtx	13 # OF CONTAINERS										

16 COMMENTS: **(Please include hazards on site.)**

17

Print Name and Company	Signature	Custody Seal # (s)	Date/Time
Sampled By:			/
Received By:			/
Relinquished By:			/
Received By:			/
Relinquished By:			/
Received By:			/
Mtx = Matrix of Sample. (AI=Air, AQ=Aqueous, LE=Leachate, ML=Misc Liquid, MS=Misc Solids, OIL, SE=Sediment, SL=Sludge, SO=Soil)			

(Copies: White and yellow copies should accompany samples to STL. The pink copy should be retained by the client.) See reverse for directions

Table 1
Maximum Constituent Concentrations in Soil
Sauget Area 2

Constituent	Site O	Site P	Site Q	Site R [A]	Site S
VOCs				5800	
benzene	30.8				
chlorobenzene	58.9		100		
ethylbenzene	167		790		450
4-methyl-2-pentanone	7.69		250		93
toluene	29.5	0.413	2400		990
1,1,1-trichloroethane	1.41				12
o-xylene			2300		
xylenes (total)	615.4	0.45			620
SVOCs				19000	
1,4-dichlorobenzene	1030	8.87	1200		
1,2-dichlorobenzene	606	3.625			
1,2,4-trichlorophenol	26.9				
naphthalene	34.6				200
2-methylnaphthalene	160				
N-nitrosodiphenylamine	50				
pentachlorophenol	1620				
phenanthrene	230				
fluoranthene	74				
pyrene	282				
butyl benzyl phthalate	3846				490
benzo(a)anthracene	121.7				
1,2,4-trichlorobenzene	65.3				
chrysene	282				
phenol		3.875			
di-n-butyl-phthalate		16.25	900		1500
di-n-octyl-phthalate					310
bis(2-ethylhexyl)phthalate			1100		20000
PCBs				4800	
Aroclor 1232	30.3				
Aroclor 1242	1871				
Aroclor 1248			70		85
Aroclor 1254			360		69
Aroclor 1260			16000		41
Dioxins					
2,3,7,8-TCDD	0.00017		0.0033		
Metals					
antimony			17900		
arsenic			0.216		
cadmium	31		152000		
chromium			3650		
copper	341		1630		139
cyanide		15			
lead		526	195000		0.392
mercury	6.3	3.9	4.9		3.5
nickel	136		371		
selenium			59.9		
silver			30.2		
thallium			0.89		
zinc	1398		9520		327

all concentrations in ppm

A - Soil sampling at Site R showed VOC concentrations ranging from .15 to 5800 ppm. SVOCs were found at levels ranging from 0.017 to 19,000 ppm. Pesticides were found at levels ranging from 0.11 to 99 ppm and PCBs were detected at levels ranging from 0.75 to 4,800 ppm. Elevated levels of arsenic, chromium, lead, nickel, and mercury were also detected in Site R soils.

Table 2. Semi-volatile Organic Analytical Parameters, Reporting Limits, and Sample Matrices

Analyte	CAS Number	Biota RL* Wet wt ug/kg	Vegetation/ Earthworm Analysis
Phenol	108-95-2	1000	X
bis-(2-Chloroethyl)ether*	111-44-4	1000	X
2-Chlorophenol*	95-57-8	1000	X
1,3-Dichlorobenzene	541-73-1	1000	X
1,4-Dichlorobenzene	106-46-7	1000	X
1,2-Dichlorobenzene	95-50-1	1000	X
2-Methylphenol*	95-48-7	1000	X
2,2'-oxybis(1-chloropropane)	108-60-1	1000	X
4-Methylphenol*	106-44-5	1000	X
N-Nitroso-di-n-propylamine	621-64-7	1000	X
Hexachloroethane*	67-72-1	1000	X
Nitrobenzene*	98-95-3	1000	X
Isophorone	78-59-1	1000	X
2-Nitrophenol	88-75-5	1000	X
2,4-Dimethylphenol*	105-67-9	1000	X
bis-(2-Chloroethoxy)methane	111-91-1	1000	X
2,4-Dichlorophenol*	120-83-2	1000	X
1,2,4-Trichlorobenzene*	120-82-1	1000	X
Naphthalene*	91-20-3	1000	X
4-Chloroaniline*	106-47-8	2000	X
Hexachlorobutadiene*	87-68-3	1000	X
4-Chloro-3-methylphenol	59-50-7	1000	X
2-Methylnaphthalene	91-57-6	1000	X
Hexachlorocyclopentadiene*	77-47-4	1000	X
2,4,6-Trichlorophenol	88-06-2	1000	X
2,4,5-Trichlorophenol	95-95-4	1000	X
2-Chloronaphthalene	91-58-7	1000	X
2-Nitroaniline	88-74-4	5000	X
Dimethylphthalate	131-11-3	1000	X
Acenaphthylene	208-96-8	1000	X
2,6-Dinitrotoluene*	606-20-2	1000	X
3-Nitroaniline	99-09-2	5000	X
Acenaphthene	83-32-9	1000	X

Table 2. Semi-volatile Organic Analytical Parameters, Reporting Limits, and Sample

Analyte	CAS Number	Biota RL* Wet wt ug/kg	Vegetation/ Earthworm Analysis
2,4-Dinitrophenol	51-28-5	1000	X
4-Nitrophenol*	100-02-7	5000	X
Dibenzofuran	132-64-9	1000	X
2,4-Dinitrotoluene*	121-14-2	1000	X
Diethyl phthalate	84-66-2	1000	X
4-Chlorophenyl phenyl ether	7005-72-3	1000	X
Fluorene*	86-73-7	1000	X
4-Nitroaniline	100-01-6	5000	X
4,6-Dinitro-2-methylphenol*	534-52-1	5000	X
N-Nitrosodiphenylamine	86-30-6	1000	X
4-Bromophenyl phenyl ether	101-55-3	1000	X
Hexachlorobenzene*	118-74-1	1000	X
Pentachlorophenol*	87-86-5	5000	X
Phenanthrene	85-01-8	1000	X
Anthracene	120-12-7	1000	X
Carbazole	86-74-8	1000	X
Di-n-butylphthalate*	84-74-2	1000	X
Fluoranthene*	206-44-0	1000	X
Pyrene*	129-00-0	1000	X
Butylbenzylphthalate	85-68-7	1000	X
3,3'-Dichlorobenzidine*	91-94-1	2000	X
Benzo(a)anthracene*	56-55-3	1000	X
Chrysene	218-01-9	1000	X
bis-(2-Ethylhexyl)phthalate*	117-81-7	1000	X
Di-n-octylphthalate	117-84-0	1000	X
Benzo(b)fluoranthene	205-99-2	1000	X
Benzo(k)fluoranthene	207-08-9	1000	X
Benzo(a)pyrene*	50-32-8	1000	X
Indeneo (1,2,3-cd) pyrene	193-39-5	1000	X
Dibenzo(a,h)anthracene	53-70-3	1000	X
Benzo(g,h,l,)perylene	191-24-2	1000	X

*The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs, using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in Table 8. The compounds with (*) are those for which the lab MDL does not meet the ecological RBC. For these compounds, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

Table 3. Pesticide Analytical Parameters, Reporting Limits, and Sample Matrices

Analyte	CAS Number	Biota RL* Wet wt ug/kg	Vegetation/ Earthworm Analysis
alpha-BHC*	319-84-6	5	X
beta-BHC*	319-85-7	5	X
delta-BHC*	319-36-8	5	X
gamma-BHC (lindane)	5-89-9	5	X
Heptachlor*	76-44-8	5	X
Aldrin*	309-00-2	5	X
Heptachlor epoxide*	1024-57-3	5	X
Endosulfan I*	959-98-8	5	X
Dieldrin*	60-57-1	5	X
4,4'-DDE	72-55-9	5	X
Endrin*	72-20-8	5	X
Endosulfan II	33213-65-9	5	X
4,4'-DDD	72-54-8	5	X
Endosulfan sulfate	1031-07-8	5	X
4,4-DDD	50-29-3	5	X
Methoxychlor	72-43-5	20	X
Endrin Ketone	53494-70-5	5	X
Endrin Aldehyde	7421-36-3	5	X
alpha-Chlordane	5103-71-9	5	X
gamma-Chlordane	5103-74-2	5	X
Toxaphene*	8001-35-2	170	X

*The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in Table 8. The compounds with (*) are those for which the lab MDL does not meet the ecological RBC. For these compounds, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

Table 4. Herbicide Analytical Parameters, Reporting Limits, and Sample Matrices

Analyte	CAS Number	Biota RL* Wet wt ug/kg	Vegetation/ Earthworm Analysis
2,4-D (herbicide)*	94-75-7	25	X
2,4,-DB	94-82-6	25	X
2,4,5-TP (Silvex)*	93-72-1	25	X
2,4,5-TP*	93-76-5	25	X
Dalapon*	75-99-0	6000	X
Dicamba*	1918-00-9	60	X
Dichloroprop	120-36-5	300	X
Dinoseb*	88-85-7	300	X
MCPA*	94-74-6	6000	X
MCPP*	93-65-2	6000	X

*The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in Table 8. The compounds with (*) are those for which the lab MDL does not meet the ecological RBC. For these compounds, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

Table 5. Dioxin and Dibenzofuran Parameters, Reporting Limits, and Sample Matrices

Analyte	CAS Number	Biota RL* Wet wt ug/kg	Vegetation/ Earthworm Analysis
2,3,7,8-TCDD*	1746-01-6	1	X
1,2,3,7,8-PeCDD	40321-76-4	5	X
1,2,3,4,7,8-HxCDD	39227-28-6	5	X
1,2,3,6,7,8-HxCDD	57653-85-7	5	X
1,2,3,7,8,9-HxCDD	19408-74-3	5	X
1,2,3,4,6,7,8-HpCDD	35822-46-9	5	X
1,2,3,4,5,6,7,8-OCDD	3268-87-9	10	X
2,3,7,8-TCDF*	51207-31-9	1	X
1,2,3,7,8-PeCDF*	57117-41-6	5	X
2,3,4,7,8-PeCDF*	57117-31-4	5	X
1,2,3,4,7,8-HxCDF	70648-26-9	5	X
1,2,3,6,7,8-HxCDF	57117-44-9	5	X
1,2,3,7,8,9-HxCDF	72918-21-9	5	X
2,3,4,6,7,8-HxCDF	60851-34-5	5	X
1,2,3,4,6,7,8-HpCDF	67562-39-4	5	X
1,2,3,4,7,8,9-HpCDF	55673-89-7	5	X
1,2,3,4,5,6,7,8-OCDF	39001-02-0	10	X
Total TCDD	41903-57-5	1	X
Total PeCDD	36088-22-9	5	X
Total HxCDD	34465-46-8	5	X
Total HpCDD	37871-00-4	5	X
Total TCDF	55722-27-5	1	X
Total PeCDF	30402-15-4	5	X
Total HxCDF	55684-94-1	5	X
Total HpCDF	38988-75-3	5	X

*The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in Table 8. For those compounds for which the lab MDL does not meet the ecological RBC, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

Table 6. Polychlorinated Biphenyls (PCBs) Analytical Parameters, Reporting Limits and

Analyte	CAS Number	Biota RL* Wet wt ug/kg	Vegetation/ Earthworm Analysis
Monochlorobiphenyls	Not applicable	10	X
Dichlorobiphenyls	Not applicable	10	X
Trichlorobiphenyls	Not applicable	10	X
Tetrachlorobiphenyls	Not applicable	20	X
Pentachlorobiphenyls	Not applicable	20	X
Hexachlorobiphenyls	Not applicable	20	X
Heptachlorobiphenyls	Not applicable	30	X
Octachlorobiphenyls	Not applicable	30	X
Nonchlorobiphenyls	Not applicable	50	X
Decachlorobiphenyls	Not applicable	50	X

*The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in Table 8. For those compounds for which the lab MDL does not meet the ecological RBC, the conservative estimate of $^1/_2$ the sample reporting limit will be used in risk-based calculations.

Table 7. Inorganic Analytical Parameters, Reporting Limits, and Sample Matrices

Analyte		CAS Number	Biota RL* Wet wt ug/kg	Vegetation/ Earthworm Analysis
Aluminum*	ICP	7429-90-5	200	X
Antimony*	GFAA	7440-36-0	20	X
Arsenic	GFAA	7440-38-2	10	X
Beryllium	ICP	7440-41-7	4	X
Cadmium	ICP	7440-43-9	5	X
Chromium	ICP	7440-47-3	10	X
Copper	ICP	7440-50-8	20	X
Lead	ICP	7439-92-1	5	X
Mercury	CVAA	7439-97-6	0.2	X
Nickel	ICP	7440-02-0	40	X
Selenium	ICP	7782-49-2	10	X
Silver	GFAA	7440-22-4	10	X
Zinc	ICP	7440-66-6	20	X

*The project reporting limits were set to achieve the risk-based concentrations required for the ecological risk assessment (Table 8). The compounds with (*) are those for which the lab must report down to their MDL to achieve the RBCs. For some metals and pathways, the project RL does not meet the ecological RBC (see Table 8). For these metals, if they are non-detected in biota, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

. Semi-volatile Organic Compounds. Ecological Risk-Based Concentrations - Terrestrial Receptors

Analyte	CAS Number	Biota RL(1) Wet wt ug/kg	Test Species	Test Species Weight (kg)	NOAEL/ LOAEL (mg/kg/d)	Ecological Receptor	Receptor Weight (kg)	Ecological Food Receptor RBC (mg/kg)	Reference/ Notes
Phenol	108-95-2	1000	mouse	0.03	523	Short tailed shrew	0.015	622	USEPA, 2001
bis-(2-Chloroethyl)ether	111-44-4	1000	rat	0.3	25	Short tailed shrew	0.015	53	ATSDR, 1989
2-Chlorophenol	95-57-8	1000	rat	0.3	50	Short tailed shrew	0.015	106	USEPA, 2001
1,3-Dichlorobenzene	541-73-1	1000							
1,4-Dichlorobenzene	106-46-7	1000	mouse	0.03	600	Short tailed shrew	0.015	714	ATSDR, 1993a
1,2-Dichlorobenzene	95-50-1	1000	rat	0.6	85.7	Short tailed shrew	0.015	216	USEPA, 2001
2-Methylphenol	95-48-7	1000	rat	0.3	30	Short tailed shrew	0.015	63	ATSDR, 1992a
2,2'-oxybis(1-chloropropane)	108-60-1	1000							
4-Methylphenol	106-44-5	1000	rat	0.3	30	Short tailed shrew	0.015	63	ATSDR, 1992a
N-Nitroso-di-n-propylamine	621-64-7	1000							
Hexachloroethane	67-72-1	1000	rat	0.3	1	Short tailed shrew	0.015	2.1	USEPA, 2001
Nitrobenzene	98-95-3	1000	rat	0.3	10	Short tailed shrew	0.015	21	ATSDR, 1990b; (2)
Isophorone	78-59-1	1000	rat	0.3	500	Short tailed shrew	0.015	1057	ATSDR, 1989d
2-Nitrophenol	88-75-5	1000	rat	0.3	283	Short tailed shrew	0.015	598	ATSDR, 1992b; (2)
2,4-Dimethylphenol	105-67-9	1000	mouse	0.03	50	Short tailed shrew	0.015	59	USEPA, 2001
bis-(2-Chloroethoxy)methane	111-91-1	1000							
2,4-Dichlorophenol	120-83-2	1000	rat	0.3	0.3	Short tailed shrew	0.015	0.63	USEPA, 2001
1,2,4-Trichlorobenzene	120-82-1	1000	rat	0.3	14.8	Short tailed shrew	0.015	31	USEPA, 2001
Naphthalene	91-20-3	1000	mouse	0.03	71.6	Short tailed shrew	0.015	85	ATSDR, 1995b
4-Chloroaniline	106-47-8	2000	rat	0.3	12.5	Short tailed shrew	0.015	26	USEPA, 2001
Hexachlorobutadiene	87-68-3	1000	rat	0.3	0.2	Short tailed shrew	0.015	0.42	ATSDR, 1994c
4-Chloro-3-methylphenol	59-50-7	1000							
2-Methylnaphthalene	91-57-6	1000							
Hexachlorocyclopentadiene	77-47-4	1000	rat	0.3	7	Short tailed shrew	0.015	15	USEPA, 2001
2,4,6-Trichlorophenol	88-06-2	1000	mouse	0.03	678	Short tailed shrew	0.015	806	ATSDR, 1990d
2,4,5-Trichlorophenol	95-95-4	1000	rat	0.3	100	Short tailed shrew	0.015	211	USEPA, 2001
2-Chloronaphthalene	91-58-7	1000							
2-Nitroaniline	88-74-4	5000							
Dimethylphthalate	131-11-3	1000							
Acenaphthylene	208-96-8	1000							
nitrotoluene	606-20-2	1000	rat	0.3	3.9	Short tailed shrew	0.015	8.2	ATSDR, 1989c
aniline	99-09-2	5000							
aphthene	83-32-9	1000	mouse	0.03	700	Short tailed shrew	0.015	832	ATSDR, 1994d
2,4-Dinitrophenol	51-28-5	5000							
4-Nitrophenol	100-02-7	5000	rat	0.3	25	Short tailed shrew	0.015	53	ATSDR, 1992b
Dibenzofuran	132-64-9	1000							
2,4-Dinitrotoluene	121-14-2	1000	rat	0.3	3.9	Short tailed shrew	0.015	8.2	ATSDR, 1989c
Diethyl phthalate	84-66-2	1000	mouse	0.03	4583	Short tailed shrew	0.015	5450	Sample et al., 1996
4-Chlorophenyl phenyl ether	7005-72-3	1000							
Fluorene	86-73-7	1000	mouse	0.03	125	Short tailed shrew	0.015	149	ATSDR, 1994d
4-Nitroaniline	100-01-6	5000							
4,6-Dinitro-2-methylphenol	534-52-1	5000	rat	0.3	2.5	Short tailed shrew	0.015	5.3	ATSDR, 1995a
N-Nitrosodiphenylamine	86-30-6	1000	rat	0.3	200	Short tailed shrew	0.015	423	ATSDR, 1993d
4-Bromophenyl phenyl ether	101-55-3	1000							
Hexachlorobenzene	118-74-1	1000	rat	0.3	0.05	Short tailed shrew	0.015	0.11	ATSDR, 1998
Pentachlorophenol	87-86-5	5000	rat	0.35	0.24	Short tailed shrew	0.015	0.53	Sample et al., 1996
Phenanthrene	85-01-8	1000							
Anthracene	120-12-7	1000	mouse	0.03	1000	Short tailed shrew	0.015	1189	ATSDR, 1994d
Carbazole	86-74-8	1000							
Di-n-butylphthalate	84-74-2	1000	mouse	0.03	62	Short tailed shrew	0.015	74	ATSDR, 1990a
Fluoranthene	206-44-0	1000	mouse	0.03	125	Short tailed shrew	0.015	149	ATSDR, 1994d
Pyrene	129-00-0	1000	mouse	0.03	75	Short tailed shrew	0.015	89	USEPA, 2001
Butylbenzylphthalate	85-68-7	1000	rat	0.3	159	Short tailed shrew	0.015	336	USEPA, 2001
3,3'-Dichlorobenzidine	* 91-94-1	2000	dog	12.7	8	red fox	4.5	10	ATSDR, 1989b
Benzo(a)anthracene	56-55-3	1000	rat	0.3	15	Short tailed shrew	0.015	32	ATSDR, 1994d
Chrysene	218-01-9	1000							
bis-(2-Ethylhexyl)phthalate	117-81-7	1000	rat	0.3	50	Short tailed shrew	0.015	106	ATSDR, 1993b
Di-n-octylphthalate	117-84-0	1000	mouse	0.03	550	Short tailed shrew	0.015	654	Sample et al., 1996
Benzo(b)fluoranthene	205-99-2	1000							
Benzo(k)fluoranthene	207-08-9	1000							
Benzo(a)pyrene	50-32-8	1000	mouse	0.03	1	Short tailed shrew	0.015	1.2	Sample et al., 1996
Indeneo (1,2,3-cd) pyrene	193-39-5	1000							
Dibenzo(a,h)anthracene	53-70-3	1000							
Benzo(g,h,i)perylene	191-24-2	1000							

Notes:

oxicological benchmarks were prioritized as follows: whole body, NOAEL and LOAEL, freshwater, similar species.

e project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs, using "J" flags, to meet ecological based concentrations (RBCs) as listed in this. The compounds with (*) are those for which the lab MDL does not meet the ecological RBC. For these compounds, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

(2) - Based on acute NOAEL/LOAEL divided by and uncertainty factor of 10.

Table 8. Pesticide Ecological Risk-Based Concentrations - Terrestrial Receptors

Analyte	CAS Number	Biota RL(1) Wet wt ug/kg	Test Species	Test Species Weight (kg)	Test Species NOAEL/LOAEL (mg/kg/d)	Receptor Species	Receptor Species Weight (kg)	Ecological Receptor Food RBC (mg/kg)	Notes/ Reference
alpha-BHC	319-84-6	5	rat	0.35	1.6	short-tailed shrew	0.015	3.5	Sample et al., 1996; (3)
beta-BHC	319-85-7	5	rat	0.35	0.4	short-tailed shrew	0.015	0.88	Sample et al., 1996
delta-BHC	319-36-8	5	rat	0.35	1.6	short-tailed shrew	0.015	3.5	Sample et al., 1996; (3)
gamma-BHC (lindane)	5-89-9	5	rat	0.35	8	short-tailed shrew	0.015	18	Sample et al., 1996
Heptachlor	76-44-8	5	mink	1	0.1	short-tailed shrew	0.015	0.29	Sample et al., 1996
Aldrin	309-00-2	5	rat	0.35	0.2	short-tailed shrew	0.015	0.44	Sample et al., 1996
Heptachlor epoxide	1024-57-3	5	mouse	0.03	0.8	short-tailed shrew	0.015	0.95	ATSDR, 1993c
Endosulfan I	959-98-8	5	rat	0.35	0.15	short-tailed shrew	0.015	0.33	Sample et al., 1996
Dieldrin	60-57-1	5	rat	0.35	0.02	short-tailed shrew	0.015	0.044	Sample et al., 1996
4,4'-DDE	72-55-9	5	mouse	0.03	34	short-tailed shrew	0.015	40	ATSDR, 1994b
Endrin	72-20-8	5	mouse	0.03	0.092	short-tailed shrew	0.015	0.11	Sample et al., 1996
Endosulfan II	33213-65-9	5							
4,4'-DDD	72-54-8	5	rat	0.3	85	short-tailed shrew	0.015	180	ATSDR, 1994b
Endosulfan sulfate	1031-07-8	5							
4,4-DDT	50-29-3	5	rat	0.35	0.8	short-tailed shrew	0.015	1.8	Sample et al., 1996
Methoxychlor	72-43-5	20	rat	0.3	10	short-tailed shrew	0.015	21	ATSDR, 1994b
Endrin Ketone	53494-70-5	5							
Endrin Aldehyde	7421-36-3	5							
alpha-Chlordane	5103-71-9	5	mouse	0.03	4.6	short-tailed shrew	0.015	5.5	Sample et al., 1996
gamma-Chlordane	5103-74-2	5	mouse	0.03	4.6	short-tailed shrew	0.015	5.5	Sample et al., 1996
Toxaphene	8001-35-2	170	rat	0.35	8	short-tailed shrew	0.015	18	Sample et al., 1996

Notes:

Ecotoxicological benchmarks were prioritized as follows: whole body, NOAEL and LOAEL, freshwater, similar species.

(1) The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs, using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in this table. The compounds with (*) are those for which the lab MDL does not meet the ecological RBC. For these compounds, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

(2) - based on acute NOAEL/LOAEL divided by an uncertainty factor of 10

(3) - based on NOAEL for BHC mixed isomers.

Table 8. Herbicide Ecological Risk-Based Concentrations - Terrestrial Receptors

Analyte	CAS Number	Biota RL* Wet wt ug/kg	Test Species	Test Species Weight (kg)	Test Species NOAEL/LOAEL (mg/kg/d)	Receptor Species	Receptor Species Weight (kg)	Ecological Receptor Food RBC (mg/kg)	Notes/ Reference
2,4-D (herbicide)	94-75-7	25	rat	0.3	1	short-tailed shrew	0.015	2.1	USEPA, 2001
2,4,-DB	94-82-6	25	dog	12.7	8	red fox	4.5	10	USEPA, 2001
2,4,5-TP (Silvex)	93-72-1	25	dog	12.7	0.75	red fox	4.5	0.97	USEPA, 2001
2,4,5-TP	93-76-5	25	rat	0.3	3	short-tailed shrew	0.015	6.3	USEPA, 2001
Dalapon	75-99-0	6000	rat	0.3	8.45	short-tailed shrew	0.015	18	USEPA, 2001
Dicamba	1918-00-9	60	rabbit	1.2	3	short-tailed shrew	0.015	9.0	USEPA, 2001
Dichloroprop	120-36-5	300							USEPA, 2001
Dinoseb	88-85-7	300	rat	0.3	1	short-tailed shrew	0.015	2.1	USEPA, 2001
MCPA	94-74-6	6000	dog	12.7	0.15	red fox	4.5	0.19	USEPA, 2001
MCPP	93-65-2	6000	rat	0.3	3	short-tailed shrew	0.015	6.3	USEPA, 2001

Notes:

Ecotoxicological benchmarks were prioritized as follows: whole body, NOAEL and LOAEL, freshwater, similar species.

(1) The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs, using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in this table. The compounds with (*) are those for which the lab MDL does not meet the ecological RBC. For these compounds, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

Table 8. Polychlorinated Biphenyls (PCBs) Ecological Risk-Based Concentrations - Terrestrial

Analyte	CAS Number	Biota RL Wet wt ug/kg	Test Species	Test Species Weight (kg)	Test Species NOAEL/LOAEL (mg/kg/d)	Receptor Species	Species Weight (kg)	Receptor Food RBC (mg/kg)	Notes/ Reference
Aroclor-1016	12674-11-2	(1)	mink	1	1.37	short-tailed shrew	0.015	3.9	Sample et al., 1996
Aroclor-1221	11104-28-2	(1)	rat	0.3	35	short-tailed shrew	0.015	74	ATSDR, 1997
Aroclor-1232	11141-16-5	(1)							
Aroclor-1242	53469-21-9	(1)	mink	1	0.069	short-tailed shrew	0.015	0.20	Sample et al., 1996
Aroclor-1248	12672-29-6	(1)	mouse	0.03	1.3	short-tailed shrew	0.015	1.5	ATSDR, 1997; (2)
Aroclor-1254	11097-79-1	(1)	oldfield mouse	0.014	0.155	short-tailed shrew	0.015	0.15	Sample et al., 1996
Aroclor-1260	11096-82-5	(1)	rat	0.3	5	short-tailed shrew	0.015	11	ATSDR, 1997

Notes:

Ecotoxicological benchmarks were prioritized as follows: whole body, NOAEL and LOAEL, freshwater, similar species.

(1) - PCB analysis will be performed using SW 846 680 which detectes PCB homologs rather than Aroclor mixtures (see Table 6). However, ecotoxicological benchmarks are generally not available for PCB homologs; therefore, RBCs were derived for Aroclor mixtures.

(2) - Based on acute NOAEL/LOAEL divided by an uncertainty factor of 10.

Table 8. Dioxin and Dibenzofuran Ecological Risk-Based Concentrations - Terrestrial Receptors

Analyte	CAS Number	Biota RL(1) Wet wt ug/kg	Test Species	Test Species Weight (kg)	Test Species NOAEL/LOAEL (mg/kg/d)	Receptor Species	Receptor Species Weight (kg)	Ecological Receptor Food RBC (mg/kg)	Notes/ Reference
2,3,7,8-TCDD	1746-01-6	1	rat	0.35	0.000001	short-tailed shrew	0.015	0.0000022	Sample et al., 1996
1,2,3,7,8-PeCDD	40321-76-4	5							
1,2,3,4,7,8-HxCDD	39227-28-6	5							
1,2,3,6,7,8-HxCDD	57653-85-7	5							
1,2,3,7,8,9-HxCDD	19408-74-3	5							
1,2,3,4,6,7,8-HpCDD	35822-46-9	5							
1,2,3,4,5,6,7,8-OCDD	3268-87-9	10							
2,3,7,8-TCDF	51207-31-9	1	mouse	0.03	0.03	short-tailed shrew	0.015	0.036	USEPA, 2001
1,2,3,7,8-PeCDF	57117-41-6	5	rat	0.35	0.00016	short-tailed shrew	0.015	0.00035	Sample et al., 1996
2,3,4,7,8-PeCDF	57117-31-4	5	rat	0.35	0.00016	short-tailed shrew	0.015	0.00035	Sample et al., 1996
1,2,3,4,7,8-HxCDF	70648-26-9	5							
1,2,3,6,7,8-HxCDF	57117-44-9	5	rat	0.35	0.00015	short-tailed shrew	0.015	0.00033	Sample et al., 1996
1,2,3,7,8,9-HxCDF	72918-21-9	5							
2,3,4,6,7,8-HxCDF	60851-34-5	5							
1,2,3,4,6,7,8-HpCDF	67562-39-4	5							
1,2,3,4,7,8,9-HpCDF	55673-89-7	5							
1,2,3,4,5,6,7,8-OCDF	39001-02-0	10							
Total TCDD	41903-57-5	1							
Total PeCDD	36088-22-9	5							
Total HxCDD	34465-46-8	5							
Total HpCDD	37871-00-4	5							
Total TCDF	55722-27-5	1							
Total PeCDF	30402-15-4	5							
Total HxCDF	55684-94-1	5							
Total HpCDF	38988-75-3	5							

Notes:

Ecotoxicological benchmarks were prioritized as follows: whole body, NOAEL and LOAEL, freshwater, similar species.

(1) The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs, using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in this table. The compounds with (*) are those for which the lab MDL does not meet the ecological RBC. For these compounds, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

Table 8. Inorganic Parameters Ecological Risk-Based Concentrations - Terrestrial Receptors

Analyte		CAS Number	Biota RL(1) Wet wt ug/kg	Test Species	Test Species Weight (kg)	Test Species NOAEL/LOAEL (mg/kg/d)	Receptor Species	Species Weight (kg)	Receptor Food RBC (mg/kg)	Notes/ Reference
Aluminum	ICP	7429-90-5	3	mouse	0.03	1.93	short tailed shrew	0.015	2.3	Sample et al., 1996
Antimony	GFAA	7440-36-0	0.2	mouse	0.03	0.125	short tailed shrew	0.015	0.15	Sample et al., 1996
Arsenic	GFAA	7440-38-2	0.2	meadow vole	0.044	1.01	short tailed shrew	0.015	1.3	Sample et al., 1996
Beryllium	ICP	7440-41-7	1	rat	0.35	66	short tailed shrew	0.015	145	Sample et al., 1996
Cadmium	ICP	7440-43-9	0.5	rat	0.303	1	short tailed shrew	0.015	2.1	Sample et al., 1996
Chromium, trivalent	ICP	7440-47-3	0.5	rat	0.5	2737	short tailed shrew	0.015	6576	Sample et al., 1996
Chromium, hexavalent				rat	0.5	3.28	short tailed shrew	0.015	7.9	Sample et al., 1996
Copper	ICP	7440-50-8	2	mink	1	11.7	short tailed shrew	0.015	33	Sample et al., 1996
Lead	ICP	7439-92-1	0.5	meadow vole	0.044	118.2	short tailed shrew	0.015	155	Sample et al., 1996
Mercury	CVAA	7439-97-6	0.02	meadow vole	0.044	0.47	short tailed shrew	0.015	0.62	Sample et al., 1996
Nickel	ICP	7440-02-0	10	rat	0.35	40	short tailed shrew	0.015	88	Sample et al., 1996
Selenium	ICP	7782-49-2	0.5	meadow vole	0.044	2.96	short tailed shrew	0.015	3.9	Sample et al., 1996
Silver	GFAA	7440-22-4	0.1	rat	0.35	222.2	short tailed shrew	0.015	488	ATSDR, 1990c
Zinc	ICP	7440-66-6	2	rat	0.35	160	short tailed shrew	0.015	352	Sample et al., 1996

Notes:

Ecotoxicological benchmarks were prioritized as follows: whole body, NOAEL and LOAEL, freshwater, similar species.

(1) The project reporting limits were set as laboratory practical quantitation limits. The lab will report lower than these reporting limits, down to their MDLs, using "J" flags, to meet ecological risk-based concentrations (RBCs) as listed in this table. The compounds with (*) are those for which the lab MDL does not meet the ecological RBC. For these compounds, the conservative estimate of 1/2 the sample reporting limit will be used in risk-based calculations.

Table 9. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Semi-volatile Organic Compound Analyses of Biota Samples

Parameter	QC Compounds	Field/Matrix Duplicate Precision[c] (RPD)	MS/MSD Precision (RPD)	Blanks	MS/MSD[ab] Accuracy (% Recovery)	Surrogate[ab] Accuracy (% Recovery)
Semivolatile Analysis	All analytes	≤ 50		≤ 5x RL for phthalates ≤ RL for all others		
	phenol		≤ 35		17-103	
	2-chlorophenol		≤ 25		23-114	
	1,4-dichlorobenzene		≤ 40		10-125	
	N-nitroso-di-n-propylamine		≤ 35		11-117	
	1,2,4-trichlorobenzene		≤ 28		17-105	
	p-chloro-m-cresol		≤ 25		25-107	
	acenaphthene		≤ 25		28-102	
	4-nitrophenol		≤ 45		10-117	
	2,4-dinitrotoluene		≤ 87		10-126	
	pentachlorophenol		≤ 44		10-120	
	pyrene		≤ 25		18-136	
	nitrobenzene-d_5					12-125
	2-fluorobiphenyl					24-118
	terphenyl-d_{14}					18-153
	phenol-d_5					10-142
	2-fluorophenol					10-118
	2,4,6-tribromophenol					14-121
	2-chlorophenol-d_4					20-130 *
	1,2-dichlorobenzene-d_4					20-130 *

Notes:

General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Method 8270C), must be followed

* Advisory Limits Only

[a] Provision for wider acceptance limits near the RL may be based on professional judgement during data review/validation

[b] As required by EPA SW846 Method 8270C, these criteria limits represent the laboratory-specific limits for accuracy and precison based on analysis of biota samples

[c] Field duplicate precision based on technical judgement and USEPA National Functional Guidelines for duplicate precision

Table 10. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Pesticide Analyses of Biota Samples

Parameter	QC Compounds	Field Matrix Duplicate Precision[c] (RPD)	MS/MSD Precision (RPD)	Blanks	MS/MSD[ab] Accuracy (% Recovery)	Surrogate[ab] Accuracy (% Recovery)
Pesticide	All analytes	≤ 50		< RL		
Analysis	gamma-BHC (lindane)		≤ 37		12-138	
	hepatachlor		≤ 38		17-138	
	aldrin		≤ 38		10-144	
	dieldrin		≤ 30		28-137	
	endrin		≤ 32		33-149	
	4,4'-DDT		≤ 26		29-134	
	tetrachloro-m-xylene					10-114
	decachlorobiphenyl					27-128
	alpha BHC		≤ 40		22-101	
	beta BHC		< 40		12-102	
	delta BHC		≤ 47		10-142	
	alpha chlordane		≤ 40		45-140	
	gamma chlordane		≤ 40		11-141	
	4,4'-DDD		≤ 50		28-134	
	4,4'-DDE		≤ 23		34-121	
	endosulfan I		≤ 40		10-141	
	endosulfan II		≤ 65		10-159	
	endosulfan sulfate		≤ 50		26-144	
	endrin aldehyde		≤ 86		10-130	
	endrin ketone		≤ 31		29-112	
	heptachlor epoxide		≤ 40		15-142	
	methoxychlor		≤ 40		24-152	
	toxaphene		≤ 50		41-126	

Notes:

General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Method 8081A), must be followed.

* Advisory limits only

[a] Provisions for wider acceptance limits near the RL may be based on professional judgment during data review/validation.

[b] As required by the EPA SW846 Methods 8000B and 8081A, these QC limits represent the laboratory-specific limits for accuracy and precision based on analysis of biota samples

[c] Field duplicate precision based on technical judgment and USEPA National Functional Guidelines for duplicate precision

Table 11. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Herbicide Analysis of Biota Samples

Parameter	QC Compounds	Field/Matrix Duplicate Precision[c] (RPD)	MS/MSD Precision (RPD)	Blanks	MS/MSD[ab] Accuracy (% Recovery)	Surrogate[ab] Accuracy (% Recovery)
Herbicide Analysis	All analytes	≤50		< RL		
	2,4-D		≤50		19-153	
	2,4-DB		≤50		20-160	
	2,4,5-TP (silvex)		≤50		27-120	
	dalapon		≤50		10-170	
	dicamba		≤50		20-160	
	2,4-dichlorophenyl acetic acid (DCAA)					30-189

Notes:

General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Method 8151A), must be followed.

[a] Provision for wider acceptance limits near the RL may be based on professional judgement during data review/validation

[b] As required by EPA SW846 Methods 8000B and 8151A, these QC limits represent the laboratory specific limits for accuracy and precision based on analysis of biota samples

[c] Field duplicate precision based on technical judgement and USEPA National Functional Guidelines for duplicate precision.

Table 12. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Dioxin and Dibenzofuran Analyses of Biota Samples

Parameter	QC Compounds	Field/Matrix Duplicate Precision (RPD)	MS/MSD Precision (RPD)	Blanks	Internal[a] Standard Accuracy (% Recovery)	Recovery Standard Accuracy
Dioxin and Dibenzofuran Analysis	All analytes	≤ 25		< RL		
	2,3,7,8-TCDD		≤ 40			
	1,2,3,7,8-TCDF		≤ 40			
	1,2,3,7,8-PeCDD		≤ 40			
	1,2,3,7,8-PeCDF		≤ 40			
	2,3,4,7,8-PeCDF		≤ 40			
	1,2,3,4,7,8-HxCDD		≤ 53			
	1,2,3,6,7,8-HxCDD		≤ 53			
	1,2,3,7,8,9-HxCDD		≤ 53			
	1,2,3,4,7,8-HxCDF		≤ 46			
	1,2,3,6,7,8-HxCDF		≤ 46			
	1,2,3,7,8,9-HxCDF		≤ 46			
	2,3,4,6,7,8-HxCDF		≤ 46			
	1,2,3,4,6,7,8-HpCDD		≤ 50			
	1,2,3,4,6,7,8-HpCDF		≤ 50			
	1,2,3,4,7,8,9-HpCDF		≤ 50			
	OCDD		≤ 50			
	OCDF		≤ 50			
	$^{13}C_{12}$-2,3,7,8-TCDD				25-150	
	$^{13}C_{12}$-2,3,7,8-TCDF				25-150	
	$^{13}C_{12}$-1,2,3,7,8-PeCDD				40-135	
	$^{13}C_{12}$-1,2,3,7,8-PeCDF				40-135	
	$^{13}C12$-1,2,3,6,7,8-HxCDD				25-150	
	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF				40-135	
	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD				25-150	
	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF				40-135	
	$^{13}C_{12}$-OCDD				25-150	
	$^{13}C_{12}$-1,2,3,4-TCDD					Method LCL +
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD					UCL criteria met

Notes:

General: All methods for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Method 8290), must be followed.
a Provision for wider acceptance limits near the RL may be based on professional judgement during data review/ validation

Table 13. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for PCB Analyses of Biota Samples

Parameter	QC Compounds	Field/Matrix Duplicate Precision (RPD)	MS/MSD Precision (RPD)	Blanks	MS/MSD[ab] Accuracy (% Recovery)	Surrogate[ab] Accuracy (%Recovery)
PCB Analysis	All analytes	≤50		< RL		
	Monochlorobiphenyls		≤50		30-130	
	Dichlorobiphenyls		≤50		30-130	
	Trichlorobiphenyls		≤50		30-130	
	Tetrachlorobiphenyls		≤50		40-140	
	Pentachlorobiphenyls		≤50		40-140	
	Hexachlorobiphenyls		≤50		40-140	
	Heptachlorobiphenyls		≤50		40-140	
	Octachlorobiphenyls		≤50		30-130	
	Nonachlorobiphenyls		≤50		30-130	
	Decachlorobiphenyls		≤50		30-130	
	Decachlorobiphenyl - $^{13}C_{12}$					27-128

Notes:

General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Method 8082), must be followed.

[a] Provisions for wider acceptance limits near the RL may be based on professional judgement during data review/ validation

[b] As required by EPA SW846 Methods 8000B and 8082, these QC limits represent the laboratory-specific limits for accuracy and precision based on analysis of biota samples. The MS/MSD should contain the most representative PCBs for the site.

[c] Field duplicate precision based on technical judgement and USEPA National Functional Guidelines for duplicate precision.

Table 14. Analytical Laboratory Data Quality Objectives for Precision and Accuracy for Inorganic Compound Analyses of Biota Samples

Parameter	QC Compounds	Field Duplicate Precision (RPD)	Sample/MD Precision (RPD)	MS Accuracy (% Recovery)	Blanks	LCS/SRM Accuracy (% Recovery)
Inorganic Analysis (metals)	All analytes	≤ 50	<20% RPD for results >5x RL; difference <± RL for results <5x RL	75-125 b, d	< ± RL	Manufacturer's Control Limits

Notes:

General: All method requirements for QC frequency and criteria for acceptance, as defined in the EPA methods for this program (SW846 Methods 6010B, 7000 series, 9010B), must be followed

[a] Provision for wider acceptance limits near the RL may be based on professional judgement during data review/validation.

[b] Unless the sample concentration exceeds the spike added concentrations by a factor of 4 or more

[c] Field duplicate precision based on technical judgement and USEPA National Functional Guidelines for duplicate precision

[d] Mercury 80-100% recovery

Table 15. Summary of QC Sample Types, Criteria and Corrective Action

Field Generated QC Samples

TYPE	PURPOSE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Field Blank (Equipment Rinsate Blanks)	Evaluate cleanliness of sample containers and sample handling and collection procedures	1 per media per 10 field samples collected	all compounds of interest < RL	Qualify Data
Field Duplicate	Evaluate precision and representativeness taking into account variability of sample matrix	1 per media per 10 field samples collected	plus 50% RPD with provisions for wider acceptance limits near the detection limits	Compare to matrix duplicates, check for possible matrix interferences or improper sample collection procedure, Qualify Data

Laboratory Generated QC Samples

TYPE	PURPOSE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS) and Standard Reference Material (SRM)	Evaluate laboratory performance (accuracy) using verified standards from an outside source	1 per media per 20 field samples or per laboratory sample batch, whichever is more frequent	Vendor supplied: Within the 95% confidence interval/ vendor supplied limits	Re-prepare and re-analyze associated samples to obtain acceptable LCS/SRM. Check if MS/MSD acceptable to compare for matrix effects.
Calibration Check Sample	Verifies calibration cruve	Minimum of 1 per analytical batch per day	90-110% recovery for inorganics; as specified in EPA methods for organics listed in Table 16	Recalibrate: check system
Method Blank	Verifies clean reagents instrument systems, and lab procedures	Minimum of 1 per analytical batch or per 20 field samples: whichever is more frequent	All compounds of interest < RL	Reanalyze; if second blank exceeds criteria, clean and recalibrate system; document corrective action

Table 15 Summary of QC Sample Types, Criteria and Corrective Action

Field Generated QC Samples

TYPE	PURPOSE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Matrix spikes and matrix duplicates MS/MSD/MD	Evaluate precision and accuracy taking into account variability of sample matrix	1 set per media per 20 field samples	Recoveries for MS/MSD specified in Tables 9 through 14 and in laboratory SOPs. RPD for sample/MS in Tables 9 through 14.	Qualify data for matrix effect if LCS/SRM is acceptable. Qualify sample/MD if precision is not acceptable. If LCS/RSM is not acceptable, see above.
Surrogate Standards	Measures recoveries in actual sample matrices	All GC/MS and all GC samples for organic analyses.	Recoveries as specified in Tables 9, 10, 11, 12 13 and in laboratory SOPs	Reanalyze samples: Qualify data.
Internal Standards	Provides standard for calculating analyte response and concentrations	All GC/MS and all GC samples for organic analyses.	Recoveries as specified in the EPA methods listed in Table 17.	Reanalyze samples: Qualify data.

RL = Reporting Limit
MS = Matrix spike sample
MSD = Matrix spike duplicate sample
MD = Matrix duplicate sample
SRM = Standard reference material
LCS = Laboratory control sample
RPD = Relative percent difference (between duplicate results)
GC = Gas Chromatography
GC/MS = Gas Chromatography/Mass Spectrometry

Note:

Qualification criteria and qualifier for each QC parameter are given in QAPP, Section 9 Data Reduction, Validation and Reporting

All additional USEPA methods QC, including initial and continuing calibration requirements and criteria for acceptance, must be followed. Laboratories will follow the method-specific corrective actions for these QC criteria, as defined in the EPA methods listed in Section 7 of this QAPP and the internal quality control checks defined in Section 8 of this QAPP.

Table 16 Biota Tissue Analyses: Number, Sample Preservation, Container Specifications, and Holding Time Requirements

Parameter	Number of Samples [a]	Sample Container	Preservative	Holding Time [b]
SVOCs	24 vegetation/24 earthworm	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
Herbicides	24 vegetation/24 earthworm	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
Pesticides	24 vegetation/24 earthworm	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
Dioxins/Dibenzofurans	24 vegetation/24 earthworm	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
PCBs	24 vegetation/24 earthworm	sealable plastic bag	Store at <-10°C (dry ice)	Extraction: 14 days after collection/thaw. Analysis: within 40 days of extraction
Metals	24 vegetation/24 earthworm	sealable plastic bag	Store at <-10°C (dry ice)	180 days - all metals except mercury. Mercury - 28 days.

(a) - Number of samples does not include QA/QC samples.
(b) - All holding times start from the time samples are thawed, if received frozen.

Table 17
Laboratory Analytical Methods for Biota
Sauget Area 2 Sites
Terrestrial Component - Ecological Risk Assessment

Parameter Type	Method of Analysis[a]
Volatile Organic Compounds	8240
Semi-volatile Organic Compounds	8270C
Pesticides	8081A
Herbicides	8151A
Dioxin and Dibenzofuran	8290 [b]
PCBs (homologs)	680
Metals	6010B (ICP)/7000 Series Methods (GFAA)/mercury-7471A

(a) - Analytical Method is from USEPA SW-846, 3rd Edition, December 1996 unless otherwise specified

(b) - SW-846, 3rd Edition, Septmber 1994 method included in the December 1996 update.

STL Savannah
Laboratory Quality Manual

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LABORATORY QUALITY MANUAL

Revision : 0
January 2000

Prepared by and for:
STL-SAVANNAH LABORATORIES

Savannah Division
5102 LaRoche Avenue
Savannah, GA 31404
(912) 354-7858

Mobile Division
900 Lakeside Drive
Mobile, AL 36693
(334) 666-6633

Tallahassee Division
2846 Industrial Plaza Drive
Tallahassee, FL 32301
(850) 878-3994

Tampa Division
6712 Benjamin Rd., Ste. 100
Tampa, FL 33634
(813) 885-7427

James W. Andrews
James W. Andrews, Ph.D.
Regional Vice President

January 28, 2000
Date

Kirstin McCracken
Kirstin McCracken
Regional QA Manager

January 28, 2000
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2.0 TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

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3.0 INTRODUCTION, PURPOSE, AND SCOPE

3.1 STL Overview

STL-Savannah Laboratories (STL-SL) is a part of Severn Trent Laboratories (STL) which is a part of Severn Trent Services Inc. (STS), a major group of US based companies. All three companies are owned by Severn Trent, plc, an international provider of water and wastewater services headquartered in Birmingham, UK.

STL-SL offers a broad range of environmental testing services provided by over three hundred professionals at four locations in the southeastern US. STL's testing capabilities include chemical, physical, and biological analyses of a variety of matrices, including aqueous, solid, drinking water, waste, tissue, air and saline/estuarine samples. Specialty capabilities include air toxics, radiological testing, tissue preparation and analysis, aquatic toxicology, asbestos, and mobile laboratory services. STL-SL facility locations and contact information are outlined in Table 1.

Table 1 STL-SL Facility Locations

Facility	Address	Telephone	Facsimile
STL-SL Savannah	5102 LaRoche Avenue Savannah, GA 31409	(912) 354-7858	(912) 352-0165
STL-SL Tallahassee	2846 Industrial Plaza Drive Tallahassee, FL 32301	(850) 878-3994	(850) 878-9504
STL-SL Mobile	900 Lakeside Drive Mobile, AL 36693	(334) 666-6633	(334) 666-6696
STL-SL Tampa	6712 Benjamin Rd., Ste. 100 Tampa, FL 33634	(813) 885-7427	(813) 885-7049

3.2 Quality Assurance Policy

It is STL-SL's policy to:

- provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- provide STL-SL clients with the highest level of professionalism and the best service practices in the industry.
- build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- maintain a working environment that fosters open communication with both clients and staff.

3.3 Management Commitment to Quality Assurance

STL-SL management is committed to providing the highest quality data and the best overall service in the environmental testing industry. To ensure that the data produced and reported by STL-SL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL-SL maintains a Quality System that is clear, effective, well communicated, and supported at all levels in the company.

STL Mission Statement

We enable our customers to create safe and environmentally favorable policies and practices, by leading the market in scientific and consultancy services. We provide this support within a customer service framework that sets the standard to which others aspire. This is achieved by people whose professionalism and development is valued as the key to success and through continued investments in science and technology.

3.4 Purpose

The purpose of the Laboratory Quality Manual (LQM) is to describe the STL-SL Quality System and to outline how that system enables all employees of STL-SL to meet the Quality Assurance (QA) policy. The LQM also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the Quality System are also defined in the LQM. This manual is based on the guidance in the STL Quality Management Plan (QMP), Revision 1, March 1999.

3.5 Scope

The requirements set forth in this document are applicable to all STL-SL facilities. The policies and practices outlined in the LQM are set forth as minimum guidelines only. Requirements that are more rigorous may be applied for specific client or regulatory programs.

STL-SL operates under the regulations and guidelines of the following federal programs:

Air Force Center for Environmental Excellence (AFCEE)
US Army Corp of Engineers, Hazardous, Toxic and Radioactive Waste (USACE HTRW)
Clean Air Act (CAA)
Clean Water Act (CWA)
Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
Navy Facilities Engineering Service Center (NFESC)
National Pollution, Discharge, and Elimination System (NPDES)
Resource Conservation and Recovery Act (RCRA)
Safe Drinking Water Act (SDWA)

STL also provides services under various state and local municipal guidelines. A current Table of Analytical Services and list of certifications for each facility is provided in STL's Corporate Statement of Qualifications.

STL-Savannah Laboratories (STL-SL) is committed to providing quality data and will endeavor to use good quality control and quality assurance practices for all field sampling and laboratory analytical procedures. It is STL-SL's policy to follow this Laboratory Quality Manual, the company's standard operating procedures (SOPs), EPA guidance for all procedures referenced in this plan, and to conform to EPA and state regulatory agency guidelines for each project reported. Changes in EPA or other regulatory procedures and guidance will be incorporated during periodic revisions of this plan and the appropriate SOPs. When allowed by the EPA guidelines, Performance-Based Methods (PBM) may be utilized. Internal laboratory and corporate audits assure adherence to the procedures of this plan and approved SOPs

This QA Plan is utilized by all STL-SL facilities. Additionally, all labs operate under the same set of standard operating procedures (SOPs), and all data are incorporated into a single Laboratory Information Management System (LIMS) network which generates common QC limits, etc., and is accessible to all employees. The QA Officers in each Division implement and administer the QA Plan. Each QA Officer is independent of production, and in their absence, the Laboratory Director will serve as the QA Officer's backup on quality issues.

Each project is directed by a single Project Manager who directs all employees involved on the project and also reviews, approves, and signs all data reports. The Project Manager is responsible for all phases of STL-SL's involvement in the project, including pre-project planning, sample bottle preparation, field sampling, computer entry of work orders, approving analytical and quality control data, final review of report, and discussion of results with client. Full-time QA officers and QC staff at each laboratory assist the Project Managers. In the absence of a Project Manager, another knowledgeable Project Manager or the Lab Director will serve as backup.

STL-SL's quality assurance procedures are designed to ensure protection of client's confidentiality and proprietary rights. Data and associated client records are stored in secure locations and all STL-SL employees sign an agreement to not divulge information to any third party without written authorization from the client.

3.6 Subcontracting

Subcontracting is arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Proof of required certifications from the subcontract facility are maintained in STL project records. Where applicable, specific QC guidelines, QAPPs, and/or SOPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements.

Project reports from external subcontract laboratories are not altered and are included in original form in the final project report provided by STL.

Subcontracting may also occur between STL facilities. Subcontracting within STL is subject to the same requirements as detailed above.

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4.0 ORGANIZATION AND RESPONSIBILITY

STL Savannah Laboratories has laboratory facilities in, and conducts field operations from, Savannah, Georgia; Tallahassee, Florida; Mobile, Alabama, and Tampa, Florida. All four facilities are structured under a common administrative, data management, and quality assurance (QA) system as outlined in Figures 4.1, 4.2, 4.3, 4.4, and 4.5.

4.1 PERSONNEL RESPONSIBILITIES

The four laboratories have a combined staff of over 240 professional and support personnel. The responsibilities of key personnel are described below. In the case of temporary absence, the direct supervisor or designee will assume the responsibilities of the absent employee or delegate the responsibility to qualified personnel.

A) Regional Vice President

1) Administer corporate policy;
2) Direct General Managers and Directors;
3) Negotiate contractual agreements; and
4) Other administrative and budgetary functions.

B) General Manager

1) Direct and provide guidance to Lab Directors;
2) Develop and maintain company-client relationships;
3) Assist Regional Vice President in establishing and carrying out corporate policy;
4) Review proposals; and
5) Other administrative and budgetary functions.

C) Administrative Director

1) Coordinate risk management and insurance program;
2) Prepare financial reports;
3) Coordinate Human Resources, Payroll and Accounts Receivable activities;
4) Review contracts and subcontracts; and
5) Coordinate activities with Corporate Commercial Director.

D) Technical Director

1) Assist corporate officers with budgetary problems;
2) Maintain equipment and facilities inventory;
3) Coordinate maintenance program;
4) Ensure compliance and establish corporate policy for safety, waste disposal and shipping of hazardous materials;
5) Coordinate purchasing;
6) Provide technical support for all divisions;
7) Coordinate QA/QC and technical activities affecting all divisions;
8) Prepare cost accounting reports;
9) Inform all laboratories about method changes; and
10) Evaluate new methodologies, instruments, and data processing hardware and software.

E) Regional Quality Assurance Manager

1) Provide technical support for all divisions;
2) Coordinate QA/QC and technical activities affecting all divisions;
3) Write SOPs and other technical documents;
4) Inform all divisions about method changes;
5) Coordinate annual audits of divisions; and
6) Coordinate compilation and implementation of the Comprehensive QA Plan.

F) Client Services Director

1) Coordinate corporate marketing efforts with Laboratory Directors, Project Directors, Project Managers, and corporate marketing group;
2) Assist Corporate in defining corporate marketing policy;
3) Coordinate proposal process; and
4) Schedule trade shows, presentations, advertising and regional marketing efforts.

G) Human Resources Manager

1) Manage Human Resources and Personnel Departments; and
2) Coordinate Office Managers, administrative staff and Human Resources activities.

H) Compliance Manager

1) Ensure that the Corporate Hazardous Waste Procedures and Waste Minimization Policies are implemented at all laboratories;

2) Review monthly hazardous waste generation reports for each laboratory;

3) Review each laboratory's hazardous waste manifests for accuracy and completeness;

4) Ensure that the Safety and Chemical Hygiene Plan is implemented at all laboratories;

5) Advise senior management of waste and safety issues that need corrective action and implement as defined.

I) Laboratory Director

1) Responsible for day-to-day operation of lab;

2) Provide Project Manager guidance;

3) Establish production priorities; and

4) Approve hiring decisions.

J) Project Manager

1) Initial contact with client on individual job tasks;

2) Prepare all work plans, schedules and manpower allocations;

3) Initiate all procurement for the projects;

4) Day-to-day direction of the project team;

5) Coordinate financial and contractual aspects of the projects;

6) Provide formatting and technical review of all reports;

7) Provide day-to-day communication with the client;

8) Exercise final review and approval on all reports and invoices for the project; and

9) Respond to post project inquiries.

K) QA Officer

1) Implement the provisions of the quality system as specified in the Corporate Quality Assurance Plan;

2) Coordinate with the Project Manager and Laboratory Managers in order to ensure that project QA is maintained;

3) Be available to discuss QA activities and results with Project Managers;

4) Prepare QA reports to management;

5) Perform periodic system audits and coordinate all external QA audits;

6) Review not-in-compliance reports and approve corrective actions;

7) Coordinate the preparation and approval of all QA plans, method SOPs, QA audit responses, data packages; and

8) Coordinate training program.

L) Laboratory Manager

1) Coordinate all production activities;

2) Work with Project Managers to ensure project objectives are met;

3) Provide guidance to Department Managers; and

4) Interview and hire technical personnel.

M) Custody Supervisor

1) Schedule bottle orders and supervise bottle prep staff;

2) Supervise custody staff;

3) Coordinate with Project Manager and Field/Sampling Supervisor on scheduling field sampling efforts;

4) Identify and document custody discrepancies and communicate with the Project Manager and client on custody problems; and

5) Supervise data management staff including computer login, data entry, report preparation, and data archiving personnel.

N) Field/Sampling Supervisor

1) Coordinate and schedule sampling crews;

2) Prepare sampling reports; and

3) Ensure sampling protocols are followed.

O) Department Manager/Supervisor/Technical Manager

1) Organize workflow in department;

2) Assure adequate inventory of reagents and equipment;

3) Ensure effective maintenance and repair of instrumentation;

4) Investigate and evaluate new methodology and equipment;

5) Ensure proper training is conducted;

6) Assure quality objectives are met for the department; and

7) Assist in preparation of SOPs.

P) Office Manager

1) Assist Laboratory Director with all administrative and financial activities;

2) Coordinate all procurement/receiving with corporate procurement department;

3) Coordinate posting of all invoices with corporate accounts receivable department;

4) Assist comptroller and laboratory directors with collection of receivables;

5) Maintain inventory of all facilities and equipment;

6) Coordinate all human resources and payroll activities; and

7) Maintain petty cash and coordinates laboratory expenditures with corporate accounting department.

Q) Analyst

1) Perform preparation and/or analysis of samples using approved procedures;

2) Calculate, check, and report data in accordance with approved procedures and the Quality Assurance Plan;

3) Perform instrument maintenance and maintain instrument logs; and

4) Maintain proper documentation of all analytical steps.

R) Lab Technicians

1) Assist analysts in sample preparation and data collection;

2) Perform routine checks for data quality objectives-surrogate recoveries, LCS/MS recoveries, initial evaluation of dilutions, internal standards areas, and method blanks;

3) Assist analysts in maintaining traceability of standards and-samples;

4) Assist analysts in preparing samples, extracts, or digests for analysis; and

5) Check samples for proper preservation and maintain department sample receipt logs and chain-of-custody.

S) Custody Technician

1) Prepare and ship sample containers and/or receive samples from clients;

2) Check sample temperature and sample integrity upon receipt;

3) Maintain and sign appropriate chain-of-custody records;

4) Distribute samples to the appropriate lab department; and

5) Composite samples for testing prior to disposal.

T) Data Technician

1) Check data packages against data quality objectives;

2) Check final results against the LIMS report; and

3) Paginate and collate all items in data packages.

U) Compliance Officer

1) Provides technical assistance in complying with corporate policies concerning safety, waste, and shipping;

2) Assists Project Managers/Lab Director in developing appropriate safety precautions for new projects;

3) Monitors collection and disposal of chemical wastes; and

4) Ensures employees comply with safety and waste disposal plans.

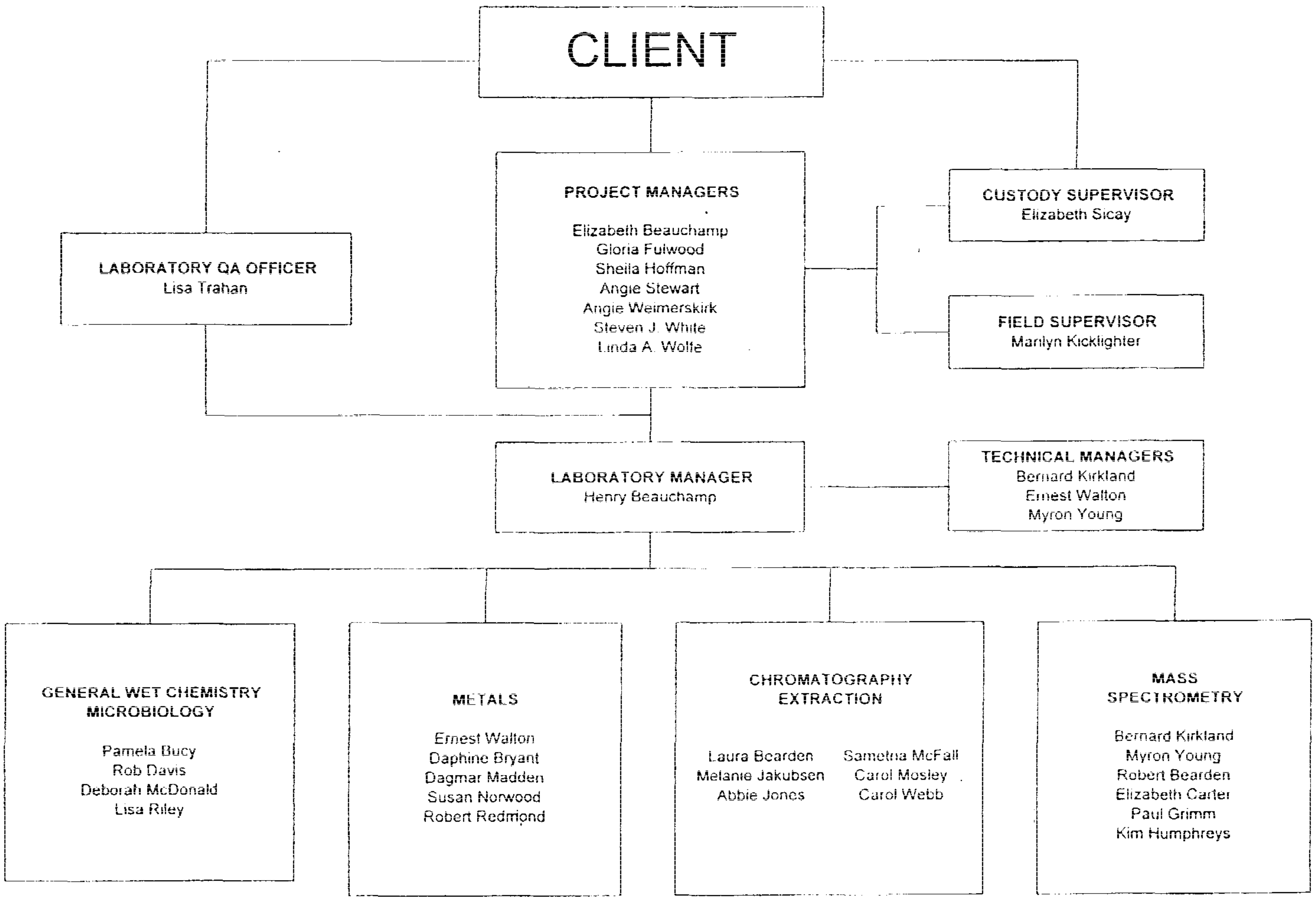
STL SAVANNAH LABORATORIES

- **REGIONAL VICE PRESIDENT** — James W. Andrews, Ph.D.
  - **GENERAL MANAGER** — Thomas L. Stephens
    - **LAB DIRECTORS**
      - TALLAHASSEE — Janet B. Pruitt
      - MOBILE — Jesse L. Smith
      - TAMPA — Andre J. Rachmaninoff
      - STL PENSACOLA — Susan Rembert
  - **CLIENT SERVICES DIRECTOR** — Janette D. Long
    - **CLIENT SERVICES**
      - PROJECT MANAGERS
      - PROJECT DIRECTORS
  - **GENERAL MANAGER** — Jack R. Tuschall, Ph.D.
    - **IT GROUP**
      - IT DIRECTOR — Wayne W. Word
      - DEPARTMENT MANAGER — Larry E. Phillips
      - TARGET SYSTEMS ADMINISTRATOR — Richie Meguiar
      - TARGET MANAGER — Bill VanAusdale
    - **LABORATORY MANAGER** — C. Henry Beauchamp
  - **TECHNICAL DIRECTOR** — Jeffrey S. Andrews
    - REGIONAL QA MANAGER — Kirstin McCracken
    - COMPLIANCE MANAGER
    - NEW TECHNOLOGY
    - PURCHASING MANAGER — Sharon Gabrielson
  - **ADMINISTRATIVE DIRECTOR** — Jay W. Andrews
    - BUSINESS MANAGER — Cathy W. Glenn
    - HUMAN RESOURCES MANAGER — Kenan J. Kern
      - OFFICE MANAGERS

STL SAVANNAH LABORATORIES
ORGANIZATIONAL STRUCTURE
Figure 4-1

11 01 9
9

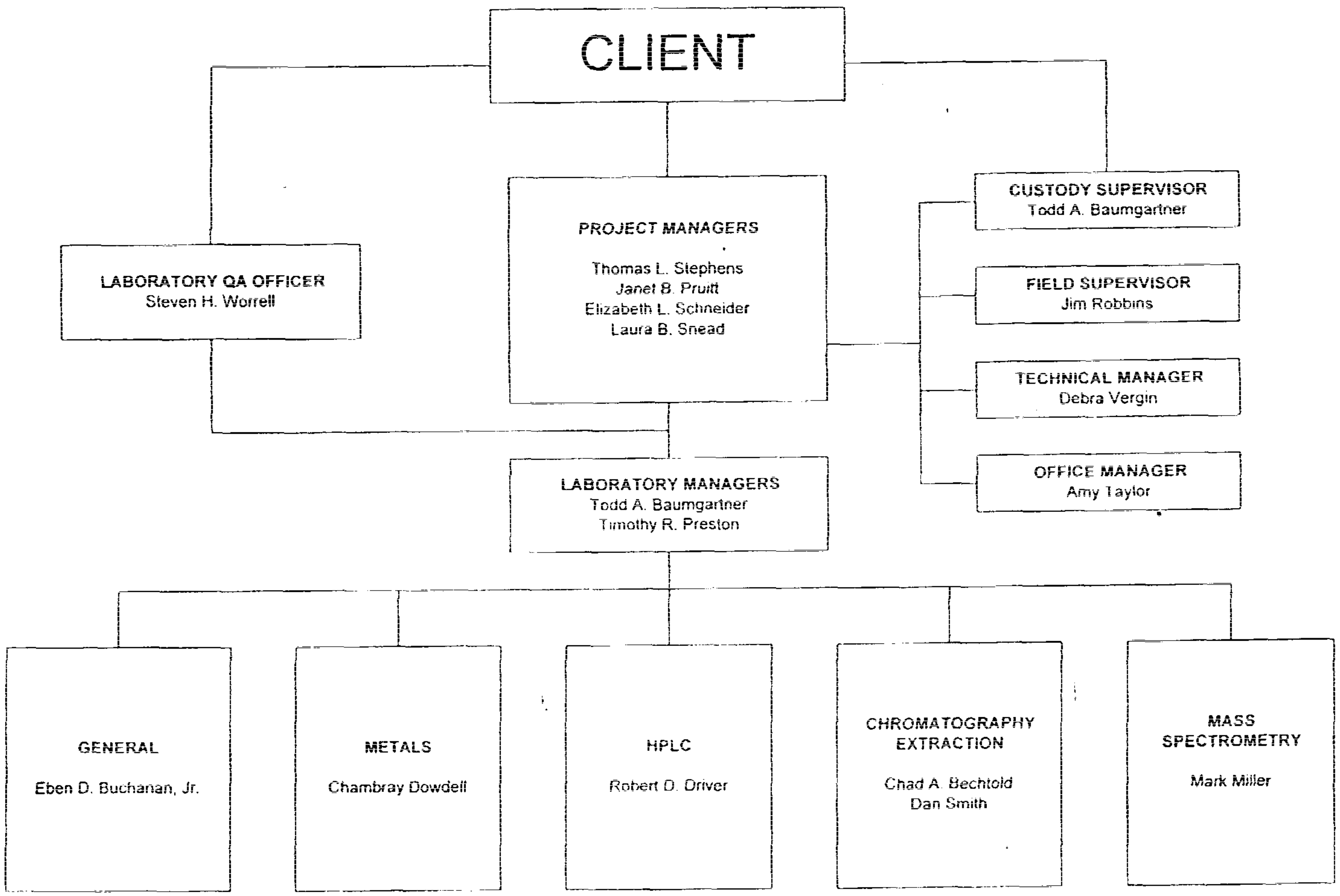
STL Savannah Laboratories
a division of Severn Trent Laboratories, Inc.



ORGANIZATIONAL STRUCTURE OF
STL SAVANNAH LABORATORIES
SAVANNAH
Figure 4-2

11 01 9
9

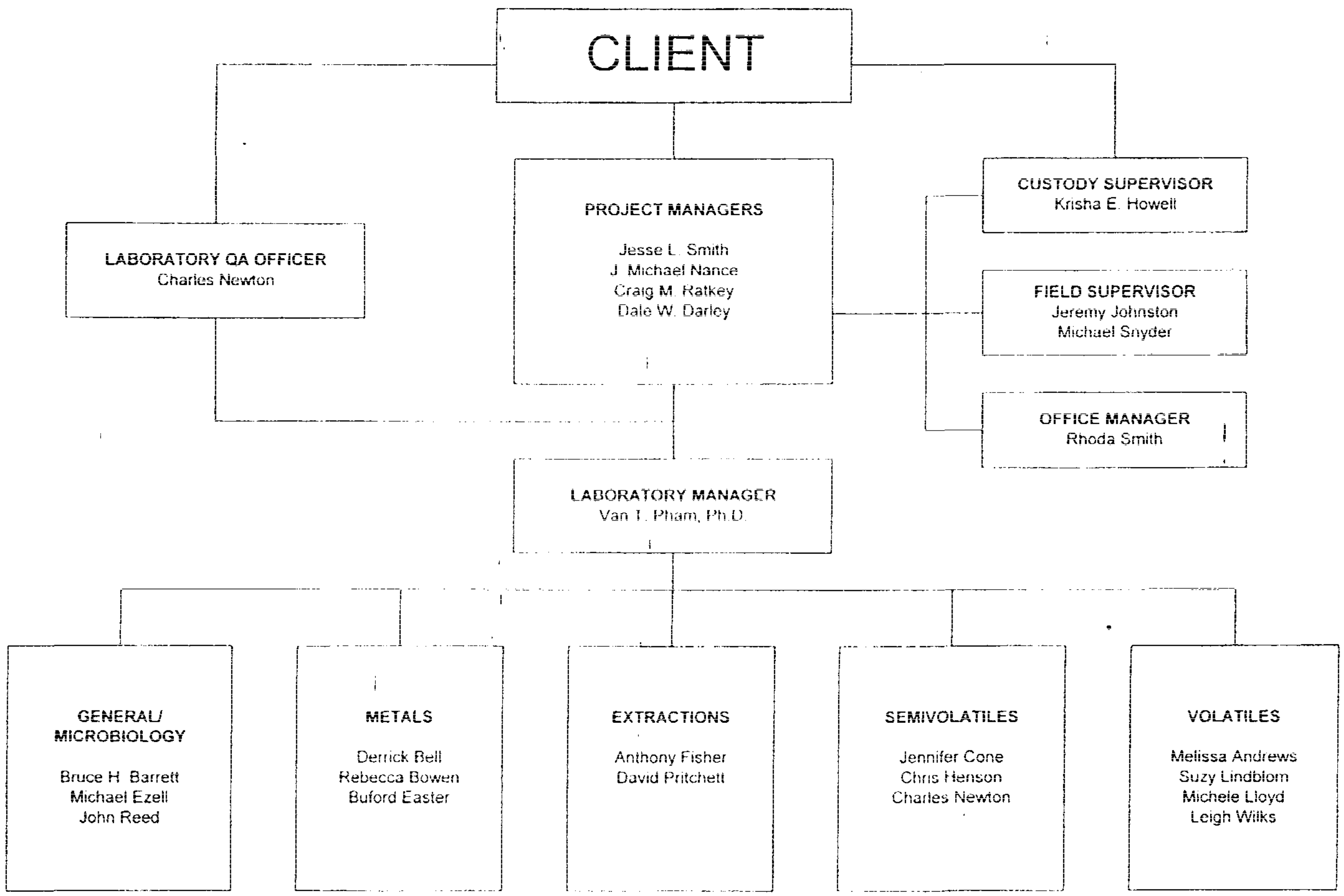
Savannah Laboratories
a division of Severn Trent Laboratories, Inc.



ORGANIZATIONAL STRUCTURE OF
STL SAVANNAH LABORATORIES
TALLAHASSEE
Figure 4-3

11 01 99

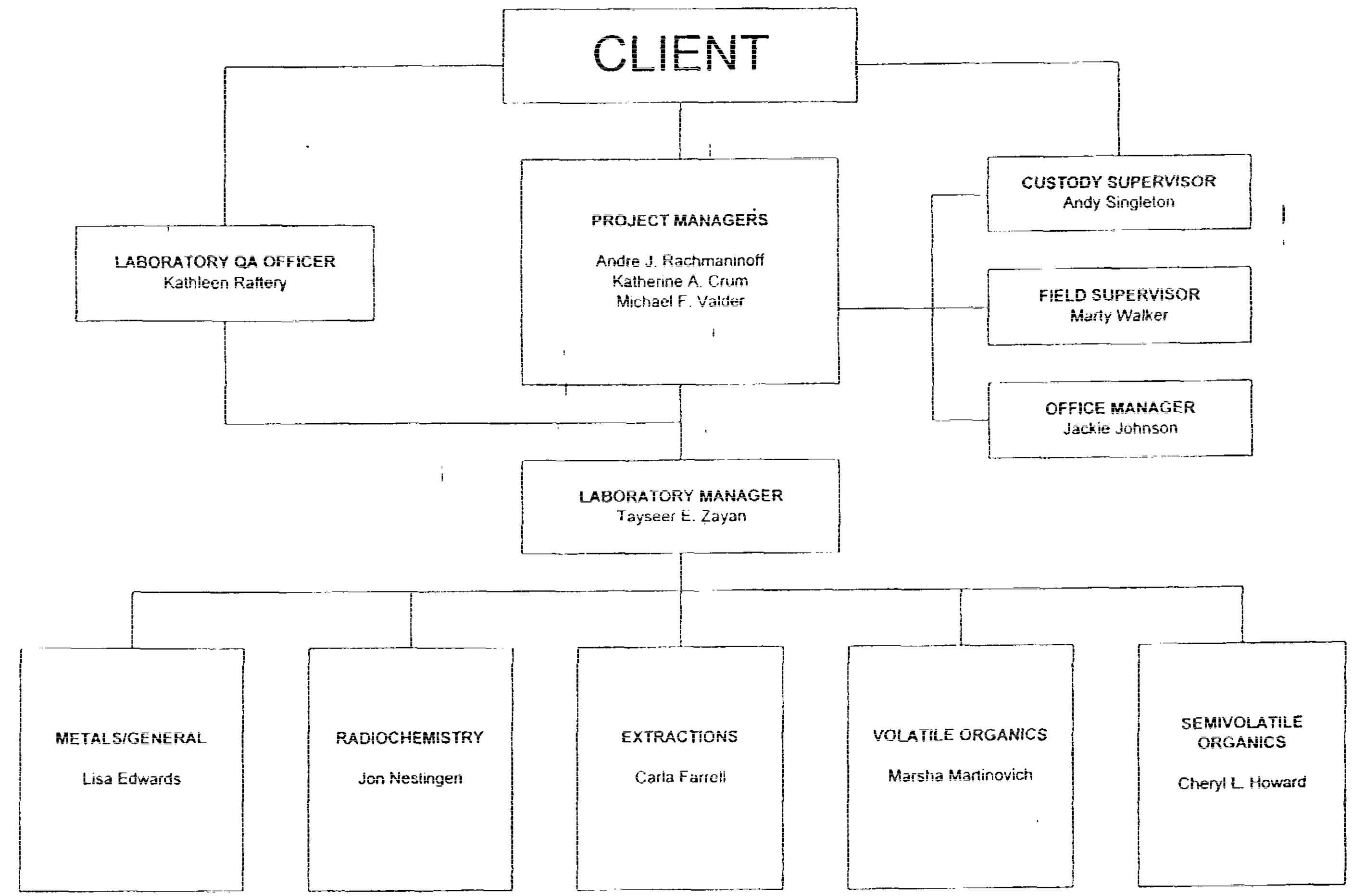
STL Savannah Laboratories
a division of Severn Trent Laboratories, Inc.



ORGANIZATIONAL STRUCTURE OF
STL SAVANNAH LABORATORIES
MOBILE
Figure 4-4

11 01 9
9

Savannah Laboratories
a division of Severn Trent Laboratories, Inc



ORGANIZATIONAL STRUCTURE OF
STL SAVANNAH LABORATORIES
TAMPA
Figure 4-5

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STL Savannah Laboratories
a division of Severn Trent Laboratories, Inc

4.2 LABORATORY FACILITIES

Each of the full-service laboratories are custom designed to accommodate modern instrumentation, permit highly efficient utilization of space, minimize employee exposure, and to reduce the potential of sample contamination. The volatile organics (VOC) analytical areas are completely isolated from the semivolatile extraction areas in order to prevent sample and blank contamination by methylene chloride, acetone, carbon disulfide and other solvent vapors. An isolated standards preparation room with a separate air conditioning/ heating system has been developed for dioxin and other hazardous substance testing.

4.3 RESOURCE COMMITMENT

State-of-the-art automation equipment is utilized, as appropriate per the methods, to operate each instrument at maximum efficiency. Instrumentation is summarized in Table 9.1. This table includes the instrument type, date, model, and date of purchase by STL Savannah Laboratories. Table 9.2 summarizes a cumulative total of instrumentation in all divisions.

4.4 COMPUTER SYSTEMS

STL-SL's operations are highly computerized in order to efficiently collect and archive data and QA results. Over 100 individual computers are used in our data generation and archiving process. The primary LIMS software runs on an NCR M3455 which is centralized at the Savannah facility and communicates with each of the laboratories via wide area network (WAN) dedicated telephone/data lines. The software utilized on the system is designed around a highly versatile UNIX/PICK relational database. Each module of the system interacts with the other modules in order to eliminate the need for re-entry of information; thus, improving production and reducing entry errors. The policies and procedures for the LIMs and other computer systems are described in the current revision of the STL Savannah Laboratories' *Software Quality Assurance Plan*.

4.5 CONTINGENCY PLAN

In case of a disaster such as a hurricane, the laboratory network would be available to provide continuity for all projects to ensure meeting sample holding time or critical project schedule requirements. In general, each facility has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation within each facility, or samples shipped to another properly certified or approved laboratory (where identical QA procedures and instruments are utilized).

The LIMS is linked to a UPS system in order to respond to power outages, hurricanes, computer failure and other disasters. In addition to this provision, a back-up computer is available at the Tallahassee, Florida facility. In case of emergency, this backup system can be accessed by the laboratories and allows all laboratories to continue functioning until the primary system is back on line.

5.0 QUALITY ASSURANCE OBJECTIVES (PRECISION, ACCURACY, AND DETECTION LIMITS)

Savannah Laboratories has a comprehensive quality assurance program which is based on the program outlined in EPA's *Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5, Interim Final August 1994, in the *Handbook for Analytical Quality Control in Water and Wastewater Laboratories* (EPA, 1979), Chapter 1, Final Updates I, II, IIB, and III of *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (SW-846), and in the Association of Official Analytical Chemists' *Quality Assurance Principles for Analytical Laboratories*.

The key to Savannah Laboratories QA/QC program is strict adherence to the program during all phases of the project including: pre-sampling discussions; sample collection, preservation, transportation and storage; sample login and tracking; laboratory analyses; and validation and reporting of results.

Project and QC data from all facilities are entered into a single Laboratory Information Management System (LIMS). The LIMS provides a computerized mechanism for storing field and login information, tracking sample holding times, scheduling and preparing laboratory work sheets, storing results and QC data, reviewing results and relating them to their corresponding QC data, and printing reports and invoices. The project manager, QA manager, and data management and reporting personnel have direct access via a CRT terminal to all project and QA data from all five facilities.

Tables 5.1 and 5.2 list the laboratory parameters determined by Savannah Laboratories, the methodology, the QA objectives for precision, accuracy and the normal method detection limits (MDLs) for relatively clean environmental samples. Table 5.3 gives the same information for TCLP samples. Tables 5.4 through 5.7 provide information for air, biological tissue, wipes and oily samples, and Table 5.8 provides information for field parameters.

PRECISION

Relative percent difference is used to express precision between two replicate values. In routine analyses, the values for most parameters are usually below quantitation limits; therefore, precision data are derived from duplicate or matrix spike duplicate results. Precision is used to evaluate matrix bias and is not used for method control, except where required in a client quality assurance project plan or agency program.

The relative percent difference (RPD) is calculated as:

$$\%RPD = \left| \frac{V1 - V2}{\left(\frac{V1 + V2}{2} \right)} \right| \otimes 100$$

V1, V2 = The two concentrations obtained by analyzing the duplicate samples, matrix spike and matrix duplicate, or lab control standard and lab control standard duplicate.

ACCURACY

Accuracy control limits are produced from environmental matrix spike data. Percent recovery (%R) is used to express accuracy. The percent recovery (%R) is calculated as below:

$$\%REC = \frac{SPV - SAV}{SA} \otimes 100$$

SPV = Value obtained by analyzing the sample with the spike added
SAV = The background value, value obtained by analyzing the unspiked sample
SA = Concentration of the spike added to the sample

COMPARABILITY

Savannah Laboratories strives for comparability of results through evaluation of data against established precision and accuracy limits. Strict adherence to QA/QC procedures promotes the comparability of one set of reference data to another or comparability of data among all facilities.

REPRESENTATIVENESS

The Savannah Laboratories objective for representativeness of field samples is to ensure that a set of data accurately depicts the chemical or biological characteristics of a sample source. Representativeness is enhanced by an attempt to mix samples prior to aliquot removal. Results are considered reliable and representative if the sample results distribution is within statistically defined bounds of the population mean and variance.

COMPLETENESS

Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. Savannah Laboratories strives to maintain 90 percent completeness. However, since completeness goals for individual projects include factors which are outside the laboratory's control (e.g., samples broken or lost in shipment, samples received outside holding times, field sampling problems, data impacted by matrix interferences), project completeness goals may not always be achieved by the laboratory. "Percent completeness" is calculated by dividing the number of valid results by the maximum number of expected results.

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Aluminum (ICP)	200.7(Drinking Water)	86	70-130	≤20	27	200
	200.7(NPDES)	3	75-125	≤20	27	200
	6010(3005/3010)	2	75-125	≤20	27	200
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	8.2	50
Antimony (ICP)	200.7(NPDES)	3	75-125	≤20	5.0	20
	6010(3005/3010)	2	75-125	≤20	5.0	20
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	1.5	5.0
Antimony (GFAA)	200.9	86	80-120	≤20	1.2	6.0
	204.2	3	80-120	≤20	1.2	10
	3113B	4	80-120	≤20	1.2	6.0
	7041(3020)	2	80-120	≤20	1.2	10
Arsenic (ICP)	200.7 (Drinking Water)	86	70-130	≤20	3.2	10
	200.7(NPDES)	3	75-125	≤20	3.2	10
	6010(3005/3010)	2	75-125	≤20	3.2	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	1.1	2.5
Arsenic (GFAA)	200.9	86	80-120	≤20	1.4	4.0
	206.2	3	80-120	≤20	2.0	10
	3113B	4	80-120	≤20	2.0	4.0
	3114B	4	80-120	≤20	0.40	2.0
	7060(3020)	2	80-120	≤20	1.5	10
	7062(3010)	2	80-120	≤20	0.40	2.0
Barium (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.2	10
	200.7(NPDES)	3	75-125	≤20	1.2	10
	6010(3005/3010)	2/3	75-125	≤20	1.2	10
	200.7/6010(3005/3010)-4X	2/3	75-125	≤20	0.094	2.5
Beryllium (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.54	4.0
	200.7(NPDES)	3	75-125	≤20	0.54	4.0
	6010(3005/3010)	2	75-125	≤20	0.54	4.0
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.014	1.0
Boron (ICP)	200.7(NPDES)	3	75-125	≤20	9.5	50
	6010(3005/3010)	2	75-125	≤20	9.5	50
Cadmium (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.71	5.0
	200.7(NPDES)	3	75-125	≤20	0.71	5.0
	6010(3005/3010)	2	75-125	≤20	0.71	5.0
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.091	1.2
Cadmium (GFAA)	200.9	86	80-120	≤20	0.16	1.0
	213.2	3	80-120	≤20	0.16	1.0
	3113B(3030E)	4	80-120	≤20	0.16	1.0
	7131(3020)	2	80-120	≤20	0.16	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug/L)	RLA (ug/L)
Calcium (ICP)	200.7(Drinking Water)	86	70-130	≤20	44	500
	200.7(NPDES)	3	75-125	≤20	44	500
	6010(3005/3010)	2	75-125	≤20	44	500
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	13	120
Chromium (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.7	10
	200.7(NPDES)	3	75-125	≤20	1.7	10
	6010(3005/3010)	2	75-125	≤20	1.7	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.25	2.5
Chromium (GFAA)	200.9	86	80-120	≤20	0.81	10
	218.2	3	80-120	≤20	0.81	10
	3113B(3030E)	4	80-120	≤20	0.81	10
	7191(3020)	2	80-120	≤20	0.81	10
Chromium, hexavalent	200.7(218.4)	3	70-130	≤20	5.0	5.0
Chromium, hexavalent	7195	2	85-115	≤20	1.4	10
(colorimetric)	7196/3500-Cr-D	2/4	85-115	≤20	1.4	10
Cobalt (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.4	10
	200.7(NPDES)	3	75-125	≤20	1.4	10
	6010(3005/3010)	2	75-125	≤20	1.4	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.12	2.5
Copper (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.90	20
	200.7(NPDES)	3	75-125	≤20	0.90	20
	6010(3005/3010)	2	75-125	≤20	0.90	20
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.53	5.0
Copper (GFAA)	220.2	3	80-120	≤20	0.67	10
	7211(3020)	2	80-120	≤20	0.67	10
	Chelation/Extraction	5	60-140	≤20	0.74	1.0
Iron (ICP)	200.7(Drinking Water)	86	70-130	≤20	18	50
	200.7(NPDES)	3	75-125	≤20	18	50
	6010(3005/3010)	2	75-125	≤20	18	50
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	3.2	12
Iron (ferrous)	3500-Fe-D (colorimetric)	4	80-120	≤20	9.4	100
Lead (ICP)	200.7(NPDES)	3	75-125	≤20	1.5	5.0
	6010(3005/3010	2	75-125	≤20	1.5	5.0
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.25	1.2
Lead (GFAA)	200.9	86	80-120	≤20	0.54	2.0
	239.2	3	80-120	≤20	0.54	5.0
	3113B(3030E)	4	80-120	≤20	0.54	2.0
	7421(3020)	2	80-120	≤20	0.54	5.0
	Chelation/Extraction	5	60-140	≤40	0.50	0.50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS						
PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug/L)	RLA (ug/L)
Lithium (ICP)	200.7(NPDES)	3	75-125	≤20	100	100
	6010(3005/3010)	2	75-125	≤20	100	100
Lithium (FLAA)	7430(3010)	2	80-120	≤20	42	100
Lithium (FLES)	SL SOP	100	80-120	≤20	0.26	2.0
Magnesium (ICP)	200.7(Drinking Water)	86	70-130	≤20	110	500
	200.7(NPDES)	3	75-125	≤20	110	500
	6010(3005/3010)	2	75-125	≤20	110	500
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	2.4	120
Manganese (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.4	10
	200.7(NPDES)	2/3	75-125	≤20	1.4	10
	6010(3005/3010)	2/3	75-125	≤20	1.4	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.082	2.5
Mercury (CVAA)	245.1	3	85-115	≤20	0.072	0.20
	7470	2	80-120	≤20	0.072	0.20
	3112B	4	80-120	≤20	0.072	0.20
Molybdenum (ICP)	200.7(Drinking Water)	86	70-130	≤20	2.6	10
	200.7(NPDES)	3	75-125	≤20	2.6	10
	6010(3005/3010)	2	75-125	≤20	2.6	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.44	2.5
Nickel (ICP)	200.7(Drinking Water)	86	70-130	≤20	4.7	40
	200.7(NPDES)	3	75-125	≤20	4.7	40
	6010(3005/3010)	2	75-125	≤20	4.7	40
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.56	10
Nickel (GFAA)	Chelation/Extraction	5	60-140	≤40	0.17	2.0
Phosphorus (ICP)	200.7(Drinking Water)	86	70-130	≤20	50	50
	6010(3010)	2	75-125	≤20	50	50
Potassium (ICP)	200.7(Drinking Water)	86	70-130	≤20	190	1000
	200.7(NPDES)	2/3	75-125	≤20	190	1000
	6010(3005/3010)	2/3	75-125	≤20	190	1000
	6010(3005/3010)-4X	86/3/2	75-125	≤20	5.7	250
Potassium (FLAA)	7610(3010)	2	80-120	≤20	100	100
	258.1	3	80-120	≤20	100	100

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug/L)	RLA (ug/L)
Selenium (ICP)	200.7(NPDES)	3	75-125	≤20	4.2	10
	6010(3005/3010)	2	75-125	≤20	4.2	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.94	2.5
Selenium (GFAA)	200.9	86	80-120	≤20	3.3	10
	270.2	3	80-120	≤20	3.3	10
	3113B(3030E)	4	80-120	≤20	3.3	10
	7740(3020)	2	80-120	≤20	3.3	10
	3114B	4	80-120	≤20	1.2	2.0
	7742(3020)	2	80-120	≤20	1.2	2.0
Silica, dissolved (ICP)	200.7(Drinking Water)	86	70-130	≤20	22	500
	200.7	3	75-125	≤20	22	500
	6010	2	75-125	≤20	22	500
Silver (ICP)	200.7(Drinking Water)	86	70-130	≤20	1.9	10
	200.7(NPDES)	3	75-125	≤20	1.9	10
	6010(3005/3010)	2	75-125	≤20	1.9	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.27	2.5
Silver(FLAA)	272.1	3	80-120	≤20	10	10
Silver(GFAA)	272.2	3	80-120	≤20	0.17	1.0
	7761(3020)	2	80-120	≤20	0.17	1.0
	Chelation/Extraction	5	60-140	≤40	0.013	0.050
Sodium (ICP)	200.7(Drinking Water)	86	70-130	≤20	310	500
	200.7(NPDES)	3	75-125	≤20	310	500
	6010(3005/3010)	2	75-125	≤20	310	500
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	10	120
Sodium (FLAA)	273.1/7770(3010)	3/2	75-125	≤20	5000	5000
Strontium (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.46	10
	200.7(NPDES)	3	75-125	≤20	0.46	10
	6010(3005/3010)	2	75-125	≤20	0.46	10
Thallium (ICP)	200.7(NPDES)	3	75-125	≤20	4.9	10
	6010(3005/3010)	2	75-125	≤20	4.9	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	1.1	2.5
Thallium (GFAA)	200.9	86	80-120	≤20	1.2	2.0
	279.2	3	80-120	≤20	1.2	10
	7841(3020)	2	80-120	≤20	1.2	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug/L)	RLA (ug/L)
Tin (ICP)	200.7(Drinking Water)	86	70-130	≤20	6.4	50
	200.7(NPDES)	3	75-125	≤20	6.4	50
	6010(3005/3010)	2	75-125	≤20	6.4	50
	200.7/6010(3005/3010)-4X	2	75-125	≤20	1.2	12
Titanium (ICP)	200.7(Drinking Water)	86	70-130	≤20	0.96	10
	200.7(NPDES)	3	75-125	≤20	0.96	10
	6010(3005/3010)	2	75-125	≤20	0.96	10
Tributyl tin (GFAA)	Atomic absorption	40	60-140	≤40	0.0010	0.0010
Vanadium (ICP)	200.7(Drinking Water)	86	70-130	≤20	2.2	10
	200.7(NPDES)	3	75-125	≤20	2.2	10
	6010(3005/3010)	2	75-125	≤20	2.2	10
	200.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	0.15	2.5
Zinc (ICP)	200.7(Drinking Water)	86	70-130	≤20	5.9	20
	200.7(NPDES)	3	75-125	≤20	5.9	20
	6010(3005/3010)	2	75-125	≤20	5.9	20
	2000.7/6010(3005/3010)-4X	86/3/2	75-125	≤20	2.6	5.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
FLAA = flame atomic adsorption spectrophotometer
FLES = flame emission spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Acetate	300.0	82	75-125	≤30	0.095	1.0
Acidity	305.1/2310B	3/4	80-120	≤30	0.40	10
	305.2	3	80-120	≤30	0.40	10
Alkalinity, total, as $CaCO_3$	310.1/2320B	3/4	80-120	≤30	0.40	1.0
Ammonia (as N)	350.1/4500-NH3-H	82/4	85-115	≤30	0.015	0.030
	350.3	82	85-115	≤30	0.011	0.050
Ammonia, un-ionized	FL-DER	60	NA	NA	NA	Calculated based on sample pH and temperature
Bicarbonate, as $CaCO_3$	4500-CO_2 D	4	NA	NA	NA	1.0
BOD-5	405.1/5210B	3/4	85-115	≤30	NA	2.0
Bromide	300.0/9056	82/2	90-110	≤30	0.068	1.0
	4110B	4	80-120	≤30	0.068	1.0
	320.1	3	75-125	≤30	0.57	2.0
Carbon, total organic	415.1/9060/5310-B	3/2/4	80-120	≤25	0.53	1.0
	5310C	4	80-120	≤25	0.54	1.0
Carbonate, as $CaCO_3$	4500-CO_2 D	4	NA	NA	NA	1.0
CBOD(1)	5210-B	4	81-119	≤30	NA	2.0
Chloride	300.0/9056	82/2	90-110	≤30	0.034	1.0
	4110B	4	80-120	≤30	0.034	1.0
	325.2/9251/4500-Cl-E	3/2/4	85-115	≤30	0.24	1.0
	325.3/9252A	3/2	75-125	≤30	0.40	1.0
	4500-Cl^- C	4	75-125	≤30	0.40	1.0
	4500-Cl^- D	4	80-120	≤30	0.40	1.0
Chlorine, residual	330.2	3	NA	≤30	NA	1.0
	330.3	3	NA	≤30	NA	1.0
	330.4	3	NA	≤30	NA	1.0
	330.5	3	NA	≤30	NA	1.0
	4500-Cl^- G	4	NA	≤40	NA	0.05
	4500-Cl^- B	4	NA	≤30	NA	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Chlorophyll	10200H	4	NA	≤30	NA	0.00010
COD	410.1	3	60-140	≤30	9.6	20
	410-2	3	60-140	≤30	9.6	20
	410.4	82	90-110	≤30	13	20
	5220C	4/71	80-120	≤40	9.6	20
	5220D	4	60-140	≤30	13	20
Coliform, fecal, MPN	9221E, C (non-potable)	4	NA	≤200	NA	2 MPN/100 mL
	9221E, C (potable)	4	NA	≤200	NA	1.1 MPN/100 mL
Coliform, fecal, MF	9222D	4	NA	≤200	NA	1 col/100 mL
Coliform, total, MPN	9131	2	NA	≤200	NA	2 MPN/100 mL
	9221B, C (non-potable)	4	NA	≤200	NA	2 MPN/100 mL
	9221B, C (potable)	4	NA	≤200	NA	1.1 MPN/100 mL
Coliform, total MF	9132	2	NA	≤200	NA	1 col/100 mL
	9222B	4	NA	≤200	NA	1 col/100 mL
Coliform, total, MMO MUG	9223/Colilert (potable)	4/81	NA	NA	NA	NA
Coliform, total, P-A	9221D, B	4	NA	NA	NA	NA
Color	110.2/2120B	3/4	NA	≤40	NA	5 PCU
	NCASI	90	75-125	≤40	NA	50 PCU
Corrosivity	2330B	4	NA	NA	NA	NA
	1110 (NACE)	2	NA	NA	NA	NA
	9040/9041	2	63-158	≤40	NA	NA
Cyanate	4500-CN-L	4	NA	NA	NA	0.030
Cyanide, amenable to chlorination	9012	2	NA	NA	NA	0.010
	335.1/9010/9014/ 4500CN⁻G	2/3/4	NA	NA	NA	0.010
Cyanide, reactive	7.3.3.2/9014	2	NA	≤50	NA	100 mg HCN/ Kg Waste
Cyanide, total	335.2/4500-CN-E	2/4	85-115	≤20	0.0050	0.010
	335.3/9012	3/2	85-115	≤20	0.0050	0.010
	335.4/9012A	82/2	90-110	≤20	0.0050	0.010
Cyanide, weak acid dissociable (WAD)	4500-CN -I	4	85-115	≤30	0.0052	0.010

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Cyanide, free	ASTM D4282-83	110	75-125	≤25	0.0050	0.010
Fluoride	300.0/9056	3/2	90-110	≤30	0.010	0.10
	4110B	4	80-120	≤25	0.010	0.10
	340.2/4500-F⁻-C (undistilled)	3/4	85-115	≤30	0.044	0.20
Formate	300.0	3	90-110	≤30	0.088	1.0
Halogens, total organic (TOX)	450.1/9020B	3/2	60-140	≤40	0.0046	0.010
Halogens, total absorbable (AOX)	1650	92	71-116	≤40	0.0047	0.020
Hardness, total (as $CaCO_3$)	2340B	4	NA	NA	NA	3.3
	130.2	3	75-125	≤30	0.40	10
Hydrazine	ASTM 1385	111	75-125	≤25	0.0050	0.10
Hydrogen ion (pH)	150.1/9040	3/2	63-158	≤40	NA	NA
Nitrate (as N)	300.0/9056	82/2	90-110	≤30	0.0090	0.10
	4110B	4	80-120	≤30	0.0090	0.10
	353.2 /4500-NO_3^-F	3/4	80-120	≤30	0.010	0.050
	353.3 /4500-NO_3^-E	3/4	80-120	≤30	0.010	0.050
Nitrate-Nitrite (as N)	353.2 /4500-NO_3^-F	3/4	80-120	≤30	0.010	0.050
	353.3 /4500-NO_3^-E	3/4	80-120	≤30	0.010	0.050
Nitrite(as N)	300.0/9056	82/2	90-110	≤30	0.0080	0.050
	4110B	4	80-120	≤30	0.0080	0.050
	353.2 /4500-NO_3^-F (w/o Cd reduction)	3/4	80-120	≤30	0.0033	0.050
	353.3 /4500-NO_3^-E (w/o Cd reduction)	3/4	80-120	≤30	0.0033	0.050
	354.1	3	80-120	≤30	0.0033	0.050
Nitrogen, total Kjeldahl (TKN)	351.2	82	75-125	≤40	0.12	0.20
Nitrogen, organic	EPA-CE: 3-205	46	NA	NA	NA	0.20
	TKN -NH3 (as N)	82/3	NA	NA	NA	0.20
Nitrogen, total	TKN + NO_2/NO_3 as (N)	2/3	NA	NA	NA	0.25
Odor	140.1/2150B	3/4	NA	NA	NA	1 TON

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Oil & Grease	413.1 /5520B	3/4	60-140	≤30	0.89	5.0
	413.2 /5520C	3/4	60-140	≤30	0.13	1.0
	1664 (HEM)	84	79-114	≤18	0.55	5.0
Orthophosphate (as P)	365.1/4500-P-F	82/4	90-110	≤30	0.0090	0.050
	365.2	3	90-110	≤30	0.0090	0.050
Oxygen, dissolved	360.1 /4500 O-G	3	NA	≤30	NA	0.10
	360.2 /4500 O-C	3/4	NA	≤30	NA	0.10
Perchlorate	300.0	113	90-110	≤30	0.00023	0.0040
Petroleum hydrocarbons	418.1	3/4	60-140	≤30	0.17	1.0
	5520F	4	60-140	≤30	0.17	1.0
	1664 (SGT-HEM)	84	66-114	≤24	1.6	5.0
Phenolics, total recoverable	420.1/ 9065	3/2	75-125	≤30	0.0090	0.050
	420.1/ 9065 (chloroform extraction)	3/2	75-125	≤30	0.0020	0.010
Phosphorus, hydrolyzable (as P)	365.1	82	60-140	≤40	0.0059	0.010
Phosphorus, organic (as P) (total minus acid hydrolyzable)	365.4	3	NA	NA	NA	0.10
Phosphorus, total (as P)	365.1	82	60-140	≤40	0.0059	0.010-
	365.2	3	60-140	≤40	0.017	0.10
	365.3	3	60-140	≤40	0.0059	0.050
	365.4 /4500P'F	3/4	60-140	≤40	0.034	0.10
Plate count, heterotrophic	9215B	4	NA	NA	NA	1000 CFU/L
Redox potential	D1498-76	38	75-125	≤20	NA	NA
Residue, dissolved	160.1/2540C	3/4	80-120	≤25	NA	5.0
Residue, suspended	160.2 / 2540D	3/4	80-120	≤25	NA	5.0
Residue, total	160.3 / 2540B	3/4	80-120	≤25	NA	5.0
Residue, volatile	160.4/2540E	3/4	NA	≤25	NA	5.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Salinity	2520B	4	NA	NA	NA	2 parts per thousand
Settleable matter	160.5/2540F	3/4	NA	NA	NA	0.20 mL
Silica, dissolved	370.1	3	75-125	≤30	2.5	10
Specific conductance	120.1/9050/2510B	3/2/4	90-110	≤10	NA	5.0 μS/cm
Specific gravity	2710F	3	NA	≤20	NA	NA
Streptococcus, fecal, MPN	9230B	4	NA	≤200	NA	2 MPN/100 mL
Streptococcus, fecal, MF	9230C	4	NA	≤200	NA	1 col/100 mL
Sulfate	300.0/9056	82/2	90-110	≤30	0.081	1.0
	4110B	4	80-120	≤30	0.081	1.0
	375.3/4500-SO_4^{2}-D	3/4	75-125	≤30	0.72	5.0
	375.4/9038/ 4500-SO_4^{2}-E	3/2/4	75-125	≤30	1.7	5.0
Sulfide	376.1 (undistilled)	3	75-125	≤30	0.40	1.0
	376.2 /4500-S^{2}-D (undistilled)	3	80-120	≤25	0.013	0.10
	9034/4500-S^{2}-E (9030-distilled)	2/4	50-150	≤50	0.80	1.0
Sulfide, reactive	7.3.4.2/9034	2	NA	≤50	NA	50 mg H_2S/ Kg Waste
Sulfite	377.1/4500-502-B	3/4	70-130	≤30	2.0	5.0
Surfactants (MBAS)	425.1/5540C	3/4	70-130	≤30	0.039	0.10
Tannins and Lignins	5550B	4	80-120	≤20	0.052	0.10
Temperature	170.1	3	NA	NA	NA	NA
Thiocyanate	4500-CN-M	4	80-120	≤25	0.023	0.10
THM formation potential	5710B	4	NA	NA	NA	0.010
Turbidity	180.1/2130B	82	90-110	≤30	0.10 NTU	0.10 NTU

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)♦♦	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL ^ (ug/L)
Benzene	502.2	51	80-120	≤22	0.27	0.50
Bromobenzene	502.2	51	80-120	≤40	0.20	0.50
Bromochloromethane	502.2	51	80-120	≤40	0.27	0.50
Bromodichloromethane	502.2	51	80-120	≤40	0.28	0.50
Bromoform	502.2	51	80-120	≤40	0.28	0.50
Bromomethane	502.2	51	60-140	≤40	0.57	1.0
n-Butylbenzene	502.2	51	60-140	≤40	0.057	0.50
sec-Butylbenzene	502.2	51	60-140	≤40	0.039	0.50
tert-Butylbenzene	502.2	51	60-140	≤40	0.045	0.50
Carbon tetrachloride	502.2	51	80-120	≤40	0.21	0.50
Chlorobenzene	502.2	51	80-120	≤29	0.10	0.50
Chloroethane	502.2	51	60-140	≤50	0.14	1.0
Chloroform	502.2	51	80-120	≤40	0.27	0.50
Chloromethane	502.2	51	60-140	≤40	0.35	1.0
2-Chlorotoluene	502.2	51	60-140	≤40	0.17	0.50
4-Chlorotoluene	502.2	51	60-140	≤40	0.21	0.50
Dibromochloromethane	502.2	51	80-120	≤40	0.22	0.50
1,2-Dibromo-3-chloropropane	502.2	51	80-120	≤40	0.25	5.0
1,2-Dibromoethane(EDB)	502.2	51	80-120	≤40	0.20	2.0
Dibromomethane	502.2	51	80-120	≤40	0.78	2.0
1,2-Dichlorobenzene	502.2	51	80-120	≤40	0.16	0.50
1,3-Dichlorobenzene	502.2	51	80-120	≤40	0.20	0.50
1,4-Dichlorobenzene	502.2	51	80-120	≤40	0.23	0.50
Dichlorodifluoromethane	502.2	51	60-140	≤50	0.62	1.0
1,1-Dichloroethane	502.2	51	80-120	≤40	0.25	0.50
1,2-Dichloroethane	502.2	51	80-120	≤40	0.20	0.50
1,1-Dichloroethene	502.2	51	80-120	≤29	0.23	0.50
cis-1,2-Dichloroethene	502.2	51	80-120	≤40	0.68	1.0
trans-1,2-Dichloroethene	502.2	51	80-120	≤40	0.29	0.50
1,2-Dichloropropane	502.2	51	80-120	≤40	0.18	0.50
1,3-Dichloropropane	502.2	51	80-120	≤40	0.22	0.50
2,2-Dichloropropane	502.2	51	80-120	≤40	0.10	0.50
1,1-Dichloropropene	502.2	51	80-120	≤40	0.081	0.50
cis-1,3-Dichloropropene	502.2	51	80-120	≤40	0.28	0.50
trans-1,3-Dichloropropene	502.2	51	80-120	≤40	0.20	0.50
Ethylbenzene	502.2	51	80-120	≤40	0.23	0.50
Hexachlorobutadiene	502.2	51	60-140	≤40	0.090	0.50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)♦♦	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Isopropylbenzene	502.2	51	60-140	≤40	0.060	0.50
4-Isopropyltoluene	502.2	51	60-140	≤40	0.052	0.50
Methylene chloride	502.2	51	80-120	≤40	1.0	1.0
Methyl t-butyl ether (MTBE)	502.2	51	80-120	≤40	1.9	2.0
Naphthalene	502.2	51	60-140	≤40	0.71	1.0
n-Propylbenzene	502.2	51	60-140	≤40	0.087	0.50
Styrene*2	502.2	51	80-120	≤40	0.057	0.50
1,1,1,2-Tetrachloroethane	502.2	51	80-120	≤40	0.46	0.50
1,1,2,2-Tetrachloroethane	502.2	51	80-120	≤40	0.18	1.0
Tetrachloroethene	502.2	51	80-120	≤40	0.14	0.50
Toluene	502.2	51	80-120	≤17	0.29	0.50
1,2,3-Trichlorobenzene	502.2	51	60-140	≤40	0.074	0.50
1,2,4-Trichlorobenzene	502.2	51	60-140	≤40	0.16	0.50
1,1,1-Trichloroethane	502.2	51	80-120	≤40	0.34	0.50
1,1,2-Trichloroethane	502.2	51	80-120	≤40	0.16	0.50
Trichloroethene	502.2	51	80-120	≤24	0.30	0.50
Trichlorofluoromethane	502.2	51	60-140	≤40	0.20	0.50
1,2,3-Trichloropropane	502.2	51	80-120	≤40	0.18	1.0
1,2,4-Trimethylbenzene	502.2	51	60-140	≤40	0.10	0.50
1,3,5-Trimethylbenzene	502.2	51	60-140	≤40	0.089	0.50
Vinyl chloride	502.2	51	60-140	≤40	0.31	1.0
o-Xylene	502.2	51	80-120	≤40	0.057	0.50
m- and p-Xylene	502.2	51	80-120	≤40	0.13	0.50
Total Xylenes	502.2	51	80-120	≤40	0.13	1.0
Surrogate - Fluorobenzene	502.2	51	50-136	NA	NA	NA
Surrogate - 2-Bromo-1-chloropropane	502.2	51	70-130	NA	NA	NA
Surrogate - 1-Bromo-3-chloropropane	502.2	51	50-136	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Chloropicrin	504	51	60-140	≤40	0.0025	0.010
1,1-Dichloropropane	504	51	60-140	≤40	0.50	2.0
1,3-Dichloropropene	504	51	60-140	≤40	0.25	1.0
Methyl isothiocyanate	504	51	60-140	≤40	5.0	20
Dibromochloropropane (DBCP)	504/504.1	91/78	70-130	≤30	0.011	0.020
Ethylene dibromide (EDB)	504/504.1	91/78	70-130	≤30	0.014	0.020
1,2,3-Trichloropropane	504.1	78	70-130	≤30	0.020	0.080

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep) ♦♦	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Alachlor	507	51	62-128	≤30	0.30	1.0
Atrazine	507	51	62-122	≤30	0.32	1.0
Bromacil	507	51	61-121	≤30	1.3	2.0
Butachlor	507	51	66-126	≤30	0.56	1.0
Disulfoton	507	51	59-119	≤60	0.66	2.0
Metalaxyl	507	51	40-160	≤30	0.56	1.0
Metolachlor	507	51	63-123	≤30	0.42	1.0
Metribuzin	507	51	71-131	≤30	0.30	1.0
Prometon	507	51	48-108	≤30	0.52	1.0
Propazine	507	51	62-122	≤30	0.26	1.0
Simazine	507	51	70-130	≤30	0.11	1.0
Surrogate - Triphenylphosphate	507	51	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)♦♦	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Aldrin	508	51	56-116	≤30	0.0030	0.010
Alpha BHC	508	51	62-122	≤30	0.0022	0.010
Beta BHC	508	51	65-125	≤30	0.0053	0.0020
Delta BHC	508	51	68-136	≤30	0.0019	0.010
Gamma BHC (Lindane)	508	51	59-119	≤18	0.0040	0.010
Alpha Chlordane	508	51	63-135	≤30	0.0028	0.010
gamma Chlordane	508	51	63-135	≤30	0.0026	0.010
Chloroneb	508	51	62-132	≤30	0.012	0.50
Chloroenzilate	508	51	78-138	≤30	0.0051	0.20
Chlorothalonil	508	51	61-121	≤30	0.0010	0.20
Dacthal (DCPA))	508	51	66-140	≤30	0.0010	0.20
4,4'-DDD	508	51	77-137	≤30	0.0060	0.020
4,4'-DDE	508	51	63-135	≤30	0.0069	0.020
4,4'-DDT	508	51	62-162	≤28	0.004	0.050
Dieldrin	508	51	57-117	≤46	0.0040	0.020
Endosulfan I	508	51	57-117	≤30	0.0027	0.020
Endosulfan II	508	51	62-122	≤30	0.0037	0.050
Endosulfan sulfate	508	51	56-148	≤40	0.0053	0.10
Endrin	508	51	58-118	≤23	0.0030	0.020
Endrin aldehyde	508	51	58-118	≤30	0.0079	0.10
Etridiazole	508	51	73-133	≤30	0.0021	0.10
Heptachlor	508	51	63-133	≤22	0.0040	0.010
Heptachlor epoxide	508	51	57-117	≤30	0.0070	0.020
Hexachlorobenzene	508	51	34-164	≤40	0.0040	0.050
Hexachlorocyclopentadiene	508	51	10-130	≤40	0.013	0.050
Methoxychlor	508	51	64-146	≤40	0.0069	0.50
Cis-Permethrin	508	51	61-121	≤30	0.0061	1.0
Trans-Permethrin	508	51	81-141	≤30	0.0038	1.0
Propachlor	508	51	73-133	≤30	0.034	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)♦♦	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Technical Chlordane	508	51	60-140	≤40	0.014	0.10
Toxaphene	508	51	60-150	≤40	0.060	1.0
Trifluralin	508	51	73-133	≤30	0.0018	0.050
PCB 1016	508	51	60-150	≤40	0.11	0.50
PCB 1221	508	51	60-150	≤40	0.25	0.50
PCB 1232	508	51	60-150	≤40	0.22	0.50
PCB 1242	508	51	60-150	≤40	0.43	0.50
PCB 1248	508	51	60-150	≤40	0.13	0.50
PCB 1254	508	51	60-150	≤40	0.12	0.50
PCB 1260	508	51	60-150	≤40	0.19	0.50
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	508	51	70-130	NA	NA	NA
Surrogate - 4,4-Dichlorobiphenyl	508	51	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep) ♦♦	REF	ACCURACY (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
2,4-D	515.1/515.2	51	49-214	≤60	0.19	0.50
Dalapon	515.1/515.2	51	48-126	≤50	0.26	10
2,4-DB	515.1/515.2	51	48-126	≤40	0.22	0.50
Dicamba	515.1/515.2	51	38-232	≤40	0.23	0.50
Dinoseb	515.1/515.2	51	DL-85	≤40	0.16	0.50
Pentachlorophenol	515.1/515.2	51	37-224	≤40	0.065	1.0
Picloram	515.1/515.2	51	45-138	≤40	0.13	0.50
2,4,5-T	515.1/515.2	51	68-166	≤62	0.020	0.50
2,4,5-TP (Silvex)	515.1/515.2	51	42-226	≤81	0.024	0.50
Surrogate - 2,4-Dichlorophenylacetic Acid (DCAA)	515.1/515.2	51	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS						
PARAMETER	METHOD (Prep) ♦♦	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL ^ (ug/L)
Benzene	524.2	51	70-130	≤30	0.090	0.50
Bromobenzene	524.2	51	70-130	≤30	0.18	1.0
Bromochloromethane	524.2	51	70-130	≤30	0.11	1.0
Bromodichloromethane	524.2	51	70-130	≤30	0.11	1.0
Bromoform	524.2	51	70-130	≤30	0.33	1.0
Bromomethane	524.2	51	70-130	≤30	0.12	2.0
n-Butylbenzene	524.2	51	70-130	≤30	0.18	1.0
Sec-Butylbenzene	524.2	51	70-130	≤30	0.12	1.0
Tert-Butylbenzene	524.2	51	70-130	≤30	0.17	1.0
Carbon tetrachloride	524.2	51	70-130	≤30	0.10	0.50
Chlorobenzene	524.2	51	70-130	≤30	0.10	0.50
Chloroethane	524.2	51	70-130	≤30	0.070	2.0
Chloroform	524.2	51	70-130	≤30	0.080	1.0
Chloromethane	524.2	51	70-130	≤30	0.080	2.0
2-Chlorotoluene	524.2	51	70-130	≤30	0.14	1.0
4-Chlorotoluene	524.2	51	70-130	≤30	0.16	1.0
Dibromochloromethane	524.2	51	70-130	≤30	0.20	1.0
1,2-Dibromo-3-chloropropane	524.2	51	70-130	≤30	0.35	2.0
1,2-Dibromoethane	524.2	51	70-130	≤30	0.10	1.0
Dibromomethane	524.2	51	70-130	≤30	0.16	1.0
1,2-Dichlorobenzene	524.2	51	70-130	≤30	0.23	0.50
1,3-Dichlorobenzene	524.2	51	70-130	≤30	0.19	1.0
1,4-Dichlorobenzene	524.2	51	70-130	≤30	0.21	0.50
Dichlorodifluoromethane	524.2	51	70-130	≤30	0.070	1.0
1,1-Dichloroethane	524.2	51	70-130	≤30	0.080	1.0
1,2-Dichloroethane	524.2	51	70-130	≤30	0.11	0.50
1,1-Dichloroethene	524.2	51	70-130	≤30	0.12	0.50
Cis-1,2-Dichloroethene	524.2	51	70-130	≤30	0.080	0.50
Trans-1,2-Dichloroethene	524.2	51	70-130	≤30	0.090	0.50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep) ♦♦	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
1,2-Dichloropropane	524.2	51	70-130	≤30	0.090	0.50
1,3-Dichloropropane	524.2	51	70-130	≤30	0.13	1.0
2,2-Dichloropropane	524.2	51	70-130	≤30	0.35	1.0
1,1-Dichloropropene	524.2	51	70-130	≤30	0.060	1.0
Cis-1,3-Dichloropropene	524.2	51	70-130	≤30	0.11	1.0
trans-1,3-Dichloropropene	524.2	51	70-130	≤30	0.14	1.0
Ethylbenzene	524.2	51	70-130	≤30	0.10	0.50
Hexachlorobutadiene	524.2	51	70-130	≤30	0.24	1.0
Isopropylbenzene	524.2	51	70-130	≤30	0.12	1.0
4-Isopropyltoluene	524.2	51	70-130	≤30	0.16	1.0
Methylene chloride	524.2	51	70-130	≤30	0.34	0.50
Methyl t-butyl ether (MTBE)	524.2	51	70-130	≤30	0.27	2.0
Naphthalene	524.2	51	70-130	≤30	0.30	1.0
n-Propylbenzene	524.2	51	70-130	≤30	0.11	1.0
Styrene	524.2	51	70-130	≤30	0.13	0.50
1,1,1,2-Tetrachloroethane	524.2	51	70-130	≤30	0.15	1.0
1,1,2,2-Tetrachloroethane	524.2	51	70-130	≤30	0.27	1.0
Tetrachloroethene	524.2	51	70-130	≤30	0.080	0.50
Toluene	524.2	51	70-130	≤30	0.13	0.50
1,2,3-Trichlorobenzene	524.2	51	70-130	≤30	0.28	1.0
1,2,4-Trichlorobenzene	524.2	51	70-130	≤30	0.26	0.50
1,1,1-Trichloroethane	524.2	51	70-130	≤30	0.080	0.50
1,1,2-Trichloroethane	524.2	51	70-130	≤30	0.17	0.50
Trichloroethene	524.2	51	70-130	≤30	0.090	0.50
Trichlorofluoromethane	524.2	51	70-130	≤30	0.050	1.0
1,2,3-Trichloropropane	524.2	51	70-130	≤30	0.34	1.0
1,2,4-Trimethylbenzene	524.2	51	70-130	≤30	0.15	1.0
1,3,5-Trimethylbenzene	524.2	51	70-130	≤30	0.15	1.0
Vinyl chloride	524.2	51	70-130	≤30	0.070	0.50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep) ♦♦	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
o-Xylene	524.2	51	70-130	≤30	0.10	0.50
m-Xylene and p-Xylene	524.2	51	70-130	≤30	0.16	0.50
Total-Xylenes	524.2	51	70-130	≤30	0.16	1.0
Surrogate - p-Bromofluorobenzene	524.2	51	70-130	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	524.2	51	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep) ♦♦	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Acenaphthene	525.2	51	70-130	≤50	0.16	0.50
Acenaphthylene	525.2	51	70-130	≤50	0.11	0.50
Anthracene	525.2	51	70-130	≤50	0.092	0.50
Benz(a)anthracene	525.2	51	70-130	≤50	0.054	0.50
Benzo(b)fluoranthene	525.2	51	35-130	≤50	0.15	0.50
Benzo(k)fluoranthene	525.2	51	35-130	≤50	0.31	0.50
Benzo(a)pyrene	525.2	51	35-130	≤50	0.13	0.20
Benzo(g,h,i)perylene	525.2	51	35-130	≤50	0.31	0.50
Butyl benzyl phthalate	525.2	51	70-130	≤50	0.42	2.0
Chrysene	525.2	51	70-130	≤50	0.11	0.50
Dibenz(a,h)anthracene	525.2	51	35-130	≤50	0.12	0.50
Di-n-butyl phthalate	525.2	51	70-130	≤50	0.19	2.0
Diethylphthalate	525.2	51	70-130	≤50	0.16	2.0
bis(2-ethylhexyl)adipate	525.2	51	35-130	≤50	0.23	2.0
bis(2-ethylhexyl)phthalate	525.2	51	35-130	≤50	1.1	2.0
Dimethylphthalate	525.2	51	70-130	≤50	0.15	2.0
Di-n-octyl phthalate	525.2	51	70-130	≤50	0.72	2.0
Fluoranthene	525.2	51	70-130	≤50	0.058	0.50
Fluorene	525.2	51	70-130	≤50	0.10	0.50
Hexachlorobenzene	525.2	51	35-130	≤50	0.10	0.50
Hexachlorocyclopentadiene	525.2	51	35-130	≤50	0.11	0.50
Indeno(1,2,3-cd)pyrene	525.2	51	35-130	≤50	0.30	0.50
Naphthalene	525.2	51	70-130	≤50	0.12	0.50
Pentachlorophenol	525.2	51	35-130	≤50	0.17	3.0
Phenanthrene	525.2	51	70-130	≤50	0.052	0.50
Pyrene	525.2	51	70-130	≤50	0.10	0.50
Surrogate-Perylene-d12	525.2	51	35-130	NA	NA	NA
Surrogate-Pyrene-d10	525.2	51	70-130	NA	NA	NA
Surrogate-1,3-Dimethyl-2-nitrobenzene	525.2	51	70-130	NA	NA	NA
Surrogate-Triphenylphosphate	525.2	51	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Aldicarb (MS)	531.1	33/51	75-138	≤20	0.15	0.50
Aldicarb sulfone	531.1	33/51	69-127	≤20	0.13	0.50
Aldicarb sulfoxide	531.1	33/51	74-136	≤40	0.13	0.50
Carbaryl	531.1	33/51	66-122	≤30	0.11	1.0
Carbofuran (MS)	531.1	33/51	71-133	≤20	0.18	1.0
3-Hydroxycarbofuran	531.1	33/51	69-127	≤20	0.17	1.0
Methiocarb	531.1	51	72-133	≤40	0.35	5.0
Methomyl	531.1	33/51	69-127	≤20	0.14	1.0
Oxamyl (MS)	531.1	33/51	68-126	≤30	0.14	1.0
Propoxur (Baygon)	531.1	33/51	76-136	≤40	0.36	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Glyphosate	547	51	53-125	≤50	29	50
Endothall	548.1	51	15-122	≤50	2.3	25
Diquat	549.1	51/56	16-120	≤50	0.42	1.0
Paraquat	549.1	51/56	27-127	≤50	0.17	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Bromodichloromethane	601	1	42-172	≤54	0.28	1.0
Bromoform	601	1	13-159	≤40	0.27	5.0
Bromomethane	601	1	D-144	≤78	0.090	1.0
Carbon tetrachloride	601	1	43-143	≤50	0.18	1.0
Chlorobenzene	601	1	38-150	≤25	0.11	1.0
Chloroethane	601	1	46-137	≤52	0.24	1.0
2-Chloroethylvinyl ether	601	1	14-186	≤50	2.5	10
Chloroform	601	1	49-133	≤36	0.11	1.0
Chloromethane	601	1	D-193	≤57	0.11	1.0
Dibromochloromethane	601	1	24-191	≤56	0.10	1.0
1,2-Dichlorobenzene	601	1	D-208	≤39	0.21	1.0
1,3-Dichlorobenzene	601	1	7-187	≤28	0.19	1.0
1,4-Dichlorobenzene	601	1	42-143	≤42	0.16	1.0
Dichlorodifluoromethane	601	1	50-130	≤50	0.23	1.0
1,1-Dichloroethane	601	1	47-132	≤25	0.13	1.0
1,2-Dichloroethane	601	1	51-147	≤58	0.12	1.0
1,1-Dichloroethene	601	1	28-167	≤35	0.13	1.0
cis-1,2-Dichloroethene(1)	601	1	38-155	≤39	0.10	1.0
Trans-1,2-Dichloroethene	601	1	38-155	≤43	0.12	1.0
Dichloromethane (Methylene chloride)	601	1	25-162	≤40	0.30	5.0
1,2-Dichloropropane	601	1	44-156	≤45	0.14	1.0
cis-1,3-Dichloropropylene	601	1	22-178	≤45	0.26	1.0
Trans-1,3-Dichloropropylene	601	1	22-178	≤55	0.14	1.0
1,1,2,2-Tetrachloroethane	601	1	8-184	≤48	0.070	1.0
Tetrachloroethylene	601	1	26-162	≤57	0.12	1.0
1,1,1-Trichloroethane	601	1	41-138	≤47	0.10	1.0
1,1,2-Trichloroethane	601	1	39-136	≤53	0.070	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Trichloroethene	601	1	35-146	≤48	0.10	1.0
Trichlorofluoromethane	601	1	21-156	≤37	0.18	1.0
Vinyl chloride	601	1	28-163	≤61	0.22	1.0
Surrogate - Bromochloromethane	601	1	70-130	NA	NA	NA
Surrogate - 2-Bromo-1-Chloropropane	601	1	70-130	NA	NA	NA
Surrogate - 1-Bromo-3-Chloropropane	601	1	70-130	NA	NA	NA
Surrogate - 1,4-Dichlorobutane	601	1	70-130	NA	NA	NA
Surrogate - 2-Bromochlorobenzene	601	1	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Benzene	602	1	39-150	≤31	0.10	1.0
Chlorobenzene	602	1	55-135	≤25	0.11	1.0
1,2-Dichlorobenzene	602	1	37-154	≤25	0.21	1.0
1,3-Dichlorobenzene	602	1	50-141	≤25	0.19	1.0
1,4-Dichlorobenzene	602	1	42-143	≤25	0.16	1.0
Ethylbenzene	602	1	32-160	≤25	0.14	1.0
Methyl Tert-Butyl Ether (MTBE)	602	1/87	40-140	≤50	0.59	10
Naphthalene	602	1/87	70-130	≤30	0.68	1.0
Toluene	602	1	46-148	≤25	0.13	1.0
Xylenes, m- and p-	602	1/87	54-125	≤30	0.27	1.0
Xylenes, o-	602	1/87	54-128	≤21	0.11	1.0
Xylenes, total	602	1/87	54-125	≤30	0.27	2.0
Surrogate - a,a,a-Trifluorotoluene	602	1	70-130	NA	NA	NA
Surrogate - 2-Bromochlorobenzene	602	1	70-130	NA	NA	NA
Surrogate - Fluorobenzene	602	1	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (%RPD)	MDL** (ug/L)	RL^ (ug/L)
Acrolein	603	1	88-118	≤30	12	20
Acrylonitrile	603	1	71-135	≤30	4.4	20
Surrogate - a,a,a-Trifluorotoluene	603	1	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
2-Chlorophenol	604	1	38-126	≤28	1.4	10
4-Chloro-3-methylphenol	604	1	49-122	≤33	1.2	10
2,4-Dichlorophenol	604	1	44-119	≤21	0.98	10
2,4-Dimethylphenol	604	1	24-118	≤30	1.7	10
2,4-Dinitrophenol	604	1	12-145	≤36	1.7	50
2-Methyl-4,6-dinitrophenol	604	1	30-136	≤33	1.4	50
2-Nitrophenol	604	1	43-117	≤26	1.4	10
4-Nitrophenol	604	1	13-110	≤33	1.1	50
Pentachlorophenol	604	1	36-134	≤28	1.4	50
Phenol	604	1	23-108	≤56	1.4	10
2,4,6-Trichlorophenol	604	1	53-119	≤30	1.4	10
Surrogate - 2,4,6-Tribromophenol	604	1	14-144	NA	NA	NA
Surrogate - 2-Flurophenol	604	1	29-121	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Bis(2-ethylhexyl) phthalate	606	1	D-158	≤82	1.3	10
Butyl benzyl phthalate	606	1	30-136	≤73	1.7	10
Diethyl phthalate	606	1	D-149	≤47	1.8	10
Dimethyl phthalate	606	1	D-156	≤63	1.1	10
Di-n-butyl phthalate	606	1	23-136	≤46	2.2	10
Di-n-octyl phthalate	606	1	D-114	≤52	1.3	10
Surrogate - 2-Fluorobiphenyl	606(FID)	1	24-160	NA	NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	606(EC)	1	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	606(EC)	1	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
N-Nitrosodimethylamine	607	1	13-109	≤38	1.7	2.0
N-Nitrosodi-n-propylamine	607	1	45-146	≤75	1.8	2.0
N-Nitrosodiphenylamine	607	1	D-139	≤67	2.1	3.0
Surrogate - Triphenylphosphate	607	1	16-164	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Aldrin	608	1	42-122	≤40	0.0099	0.050
alpha BHC	608	1	37-134	≤28	0.0079	0.050
beta BHC	608	1	17-147	≤25	0.0094	0.050
delta BHC	608	1	19-140	≤25	0.012	0.050
gamma BHC (Lindane)	608	1	32-127	≤44	0.0074	0.050
technical Chlordane	608	1	45-119	≤30	0.043	0.50
4,4'-DDD	608	1	31-141	≤25	0.018	0.10
4,4'-DDE	608	1	30-145	≤25	0.014	0.10
4,4'-DDT	608	1	25-160	≤40	0.017	0.10
Dieldrin	608	1	36-146	≤35	0.012	0.10
Endosulfan I	608	1	45-153	≤25	0.0094	0.050
Endosulfan II	608	1	D-202	0.018	0.0013	0.10
Endosulfan sulfate	608	1	26-144	≤25	0.020	0.10
Endrin	608	1	30-147	≤66	0.014	0.10
Endrin aldehyde	608	1	49-169	≤58	0.021	0.10
Heptachlor	608	1	34-111	≤43	0.0062	0.050
Heptachlor epoxide	608	1	37-142	≤25	0.0069	0.050
Toxaphene	608	1	41-126	≤30	1.0	5.0
PCB 1016	608	1	50-114	≤30	0.21	1.0
PCB 1221	608	1	15-178	≤30	0.36	2.0
PCB 1232	608	1	10-215	≤30	0.095	1.0
PCB 1242	608	1	39-150	≤30	0.20	1.0
PCB 1248	608	1	38-158	≤30	0.13	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
PCB 1254	608	1	29-131	≤30	0.22	1.0
PCB 1260	608	1	8-127	≤30	0.11	1.0
Surrogate - Dibutylchlorendate (DBC)	608	1	30-150	NA	NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	608	1	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	608	1	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Chloroneb	608.1	10	49-125	≤30	0.091	0.40
Chloropropylate	608.1	10	51-125	≤30	0.070	0.50
Chlorobenzilate (MS)	608.1	10	53-125	≤30	0.17	0.50
Etridiazole	608.1	10	60-125	≤30	0.0018	0.020
PCNB	608.1	10	60-125	≤30	0.0033	0.60
Propachlor	608.1	10	51-125	≤30	0.020	0.50
Chlorothalonil	608.2	57	55-125	≤30	0.016	0.20
DCPA (Dacthal)	608.2	57	50-150	≤40	0.0031	0.50
Dichloran	608.2	57	56-110	≤40	0.017	5.0
Methoxychlor	608.2	57	50-140	≤40	0.0047	0.50
Permethrin	608.2	57	50-130	≤40	0.081	1.0
Surrogate - Dibutylchlorendate (DBC)	608.1/608.2	10/57	30-150	NA	NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	608.1/608.2	10/57	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	608.1/608.2	10/57	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
2,4-Dinitrotoluene	609(ECD)	1	6-125	≤40	0.075	0.30
2,6-Dinitrotoluene	609(ECD)	1	8-126	≤40	0.075	0.30
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	609(ECD)	1	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	609(ECD)	1	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Gas Chromatography (FID)						
Acenaphthene	610	1	D-124	≤25	0.84	10
Acenaphthylene	610	1	D-139	≤30	0.83	10
Anthracene#1	610	1	D-126	≤28	0.84	10
Benzo(a)anthracene#2	610	1	12-135	≤38	2.4	10
Benzo(a)pyrene	610	1	D-128	≤40	0.88	10
Benzo(b)fluoranthene#3	610	1	6-150	≤51	2.6	10
Benzo(k)fluoranthene#3	610	1	6-150	≤51	2.6	10
Benzo(g,h,i)perylene	610	1	D-116	≤61	0.74	10
Chrysene#2	610	1	12-135	≤38	2.4	10
Fluoranthene	610	1	14-123	≤25	0.85	10
Fluorene	610	1	D-142	≤25	0.87	10
Indeno(1,2,3-cd) pyrene#4	610	1	D-116	≤52	0.79	10
Dibenzo(a,h)anthracene#4	610	1	D-116	≤52	0.79	10
1-Methylnaphthalene	610	1	10-145	≤50	1.4	10
2-Methylnaphthalene	610	1	14-138	≤50	1.6	10
Naphthalene	610	1	D-122	≤28	0.78	10
Phenanthrene#1	610	1	D-126	≤28	0.84	10
Pyrene	610	1	D-140	≤28	1.2	10
Surrogate- 2-Fluorobiphenyl	610	1	10-130	NA	NA	NA
Surrogate-o-Terphenyl	610	1	38-156	NA	NA	NA

*# = where the number is the same for any 2 compounds, these compounds cannot be routinely resolved chromatographically and are therefore reported as a combined result.

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/L)	RLA (ug/L)
HPLC (UV and Fluroresence)						
Acenaphthene	610	1	D-124	≤25	0.11	1.0
Acenaphthylene	610	1	D-139	≤30	0.11	1.0
Anthracene	610	1	D-126	≤28	0.0049	0.20
Benzo(a)anthracene	610	1	12-135	≤38	0.0061	0.20
Benzo(b)fluoranthene	610	1	6-150	≤51	0.0037	0.20
Benzo(k)fluoranthene	610	1	6-150	≤51	0.0011	0.20
Benzo(g,h,i)perylene	610	1	D-116	≤61	0.013	0.50
Benzo(a)pyrene	610	1	D-128	≤40	0.0075	0.20
Chrysene	610	1	12-135	≤40	0.0066	0.20
Dibenzo(a,h)anthracene	610	1	D-116	≤52	0.036	0.20
Fluoranthene	610	1	14-123	≤25	0.014	0.50
Fluorene (MS)	610	1	D-142	≤25	0.020	0.50
Indeno(1,2,3-cd)pyrene	610	1	D-116	≤52	0.011	0.20
Naphthalene	610	1	D-122	≤28	0.056	1.0
Phenanthrene	610	1	D-126	≤28	0.013	0.20
Pyrene	610	1	D-140	≤28	0.012	0.50
Surrogate-Terphenyl-d14	610	1	32-141	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/L)	RLA (ug/L)
Bis(2-chloroethoxy) methane	611	1	12-128	≤50	1.0	5.0
Bis(2-chloroethyl)ether	611	1	11-152	≤50	1.8	20
Bis(2-chloroisopropyl) ether	611	1	9-165	≤50	2.5	10
4-Bromophenyl phenyl ether	611	1	D-189	≤50	1.3	5.0
4-Chlorophenyl phenyl ether	611	1	D-170	≤50	8.8	40
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	611	1	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	611	1	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
2-Chloronaphthalene	612	1	9-148	≤50	1.4	10
1,2-Dichlorobenzene	612	1	9-160	≤50	0.20	10
1,3-Dichlorobenzene	612	1	D-150	≤50	0.24	10
1,4-Dichlorobenzene	612	1	13-137	≤50	0.57	10
Hexachlorobenzene	612	1	15-159	≤50	0.0032	0.10
Hexachlorobutadiene	612	1	D-139	≤50	0.0084	0.10
Hexachlorocylcopentadiene	612	1	D-111	≤50	0.0030	0.10
Hexachloroethane	612	1	8-139	≤50	0.0038	0.10
1,2,4-Trichlorobenzene	612	1	5-149	≤50	0.060	1.0
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	612	1	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	612	1	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) (MS)	613	1	63-137	≤40	0.00060	0.0050
Internal Standard - $^{13}C_{12}$-2,3,7,8-TCDD	613	1	> 50	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Aspon	622.1	8	62-104	≤40	0.25	1.0
Azinphos methyl (Guthion)	614/622	52/14	48-162	≤50	0.16	1.0
Bolstar	622	14	36-114	≤40	0.17	1.0
Chlorpyrifos	622	14	49-109	≤40	0.17	1.0
Chlorpyrifos methyl	622	14	53-136	≤40	0.43	1.0
Coumaphos	622	14	61-139	≤40	0.19	1.0
Demeton	622	14	10-117	≤40	1.1	2.5
Demeton-o	614	52	10-117	≤40	1.1	2.5
Demeton-s	614	52	37-121	≤40	0.22	2.5
Diazinon (MS)	614/622	52/14	40-137	≤40	0.16	1.0
Dichlofenthion	622.1	8	62-104	≤40	0.25	1.0
Dichlorvos	622	14	11-158	≤40	0.28	2.0
Dioxathion	614.1	58	76-127	≤40	2.5	10
Disulfoton	614/622	52/14	42-112	≤66	0.17	2.0
EPN	614.1	58	48-124	≤40	0.19	1.0
Ethion	614/614.1	52/58	62-175	≤40	0.11	0.50
Ethoprop	622	14	42-123	≤40	0.16	0.50
Famphur	622.1	8	13-128	≤60	0.89	2.0
Fenamiphos	614	52	40-160	≤40	0.59	2.0
Fenitrothion	622.1	8	61-103	≤40	0.50	2.0
Fensulfothion	622	14	31-163	≤40	0.25	5.0
Fenthion	622	14	41-115	≤60	0.20	1.0
Fonophos	622.1	8	53-133	≤40	0.25	1.0
Isofenphos	614	52	40-160	≤40	0.060	0.50
Malathion	614	52	10-140	≤40	0.066	1.0
Merphos	622	14	50-130	≤40	0.22	1.0
Metalaxyl	614	52	38-141	≤72	0.54	1.0
Metribuzin	614	52	75-177	≤20	0.11	2.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Mevinphos	622	14	24-166	≤40	0.17	2.0
Naled	622	14	10-119	≤40	0.070	5.0
Parathion, ethyl (MS)	614	52	28-155	≤34	0.066	2.0
Parathion, methyl (MS)	614/622	52/14	38-149	≤32	0.19	0.50
Phorate	622	14	29-119	≤40	0.12	1.0
Phosmet	622.1	8	50-150	≤40	0.25	1.0
Ronnel (MS)	622	14	30-98	≤35	0.27	1.0
Stirophos (Tetrachlorvinphos)	622	14	48-125	≤40	0.39	1.0
Terbufos	614.1	58	40-160	≤40	0.11	0.50
Thionazin (MS)	622.1	8	12-139	≤60	0.13	1.0
Tokuthion (Prothiofos)	622	14	45-114	≤40	0.22	1.0
Trichloronate	622	14	16-123	≤40	0.11	1.0
Surrogate – Triphenylphosphate	614/622.1/622	52/8/14	16-164	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
2,4-D	615	53	20-163	≤65	0.19	0.50
Dalapon	615	53	10-160	≤80	0.43	120
2,4-DB	615	53	40-140	≤50	0.23	0.50
Dicamba	615	53	10-317	≤86	0.12	1.2
Dichlorprop	615	53	10-258	≤103	0.12	6.0
Dinoseb	615	53	10-143	≤157	0.080	6.0
MCPA	615	53	10-231	≤91	15	120
MCPP	615	53	10-210	≤91	12	120
2,4,5-T	615	53	31-156	≤54	0.020	0.50
2,4,5-TP(Silvex)	615	53	41-135	≤45	0.024	0.50
Surrogate - 2,4-Dichlorophenylacetic acid (DCAA)	615	53	27-133	NA	NA	NA
Surrogate - 2,4-Dichorophenoxybutyric acid (2,4-DB)	615	53	40-140	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Captan (MS)	617	26	55-125	≤40	0.016	0.10
Carbophenothion	617	26	50-110	≤40	0.17	1.0
Dichloran	617	26	56-110	≤40	0.071	5.0
Dicofol	617	26	55-115	≤40	0.0067	0.10
Isodrin (MS)	617	26	55-110	≤40	0.0089	0.050
Mirex	617	26	54-104	≤40	0.027	0.50
PCNB	617	26	54-100	≤40	0.0033	0.020
Pendimethalin	617	26	75-146	≤20	0.0021	0.020
Perthane	617	26	55-115	≤40	1.2	5.0
Strobane	617	26	48-127	≤40	0.50	2.0
Trifluralin	617	26	54-124	≤40	0.0067	0.025
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	617	26/27	10-110	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Chloropicrin	618	27	62-134	≤40	0.25	1.0
Ethylene dibromide	618	27	48-90	≤40	0.12	0.50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Alachlor	619	7	45-140	≤30	0.14	2.0
Ametryn	619	7	60-120	≤40	0.16	2.0
Atraton	619	7	50-115	≤40	0.086	5.0
Atrazine (MS)	619	7	40-125	≤30	0.13	2.0
Benoxacor	619	7	10-150	≤50	4.4	10
Bromacil	619	7	55-127	≤30	0.24	2.0
Chlordimeform (Galecron)	619	7	10-150	≤50	3.8	10
5-Chloroaminotoluene	619	7	10-150	≤50	3.4	10
Hexazinone	619	7	50-130	≤30	0.72	2.0
Metalachlor	619	7	39-125	≤27	0.11	1.0
Metalaxyl	619	7	50-130	≤40	0.25	1.0
Metribuzin	619	7	61-141	≤30	0.11	2.0
Norflurazon	619	7	54-134	≤30	0.24	2.0
Prometon	619	7	55-124	≤40	0.11	2.0
Prometryn	619	7	55-120	≤40	0.080	2.0
Propazine (MS)	619	7	32-127	≤20	0.080	2.0
Secbumeton	619	7	30-130	≤45	0.042	5.0
Simetryn	619	7	50-200	≤40	0.059	2.0
Simazine	619	7	25-174	≤50	0.19	2.0
Terbuthylazine	619	7	60-130	≤40	0.093	2.0
Terbutryn	619	7	53-113	≤40	0.066	2.0
Triadimefon	619	7	61-125	≤30	0.31	2.0
Diphenylamine	620	23	56-125	≤30	0.50	2.0
Surrogate - Triphenylphosphate	619/620	7/23	16-164	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Acrolein	624	2	54-145	≤74	43	100
	624 (low level)	2	54-145	≤74	9.2	20
Acrylonitrile	624	2	10-183	≤41	9.7	100
	624 (low level)	2	10-183	≤41	5.0	20
Benzene	624	1	37-151	≤31	0.27	5.0
	624(low level)	1	37-151	≤31	0.10	1.0
Bromodichloromethane	624	1	35-155	≤30	0.35	5.0
	624(low level)	1	35-155	≤30	0.14	1.0
Bromoform	624	1	45-169	≤30	0.58	5.0
	624(low level)	1	45-169	≤30	0.24	1.0
Bromomethane	624	1	D-242	≤32	2.5	10
	624(low level)	1	D-242	≤32	0.44	1.0
Carbon tetrachloride	624	1	70-140	≤25	0.42	5.0
	624(low level)	1	70-140	≤25	0.090	1.0
Chlorobenzene	624	1	37-160	≤25	0.63	5.0
	624(low level)	1	37-160	≤25	0.090	1.0
Chloroethane	624	1	14-230	≤36	1.6	10
	624(low level)	1	14-230	≤36	0.22	1.0
2-Chloroethyl vinyl ether	624	1	D-305	≤96	11	50
	624(low level)	1	D-305	≤96	0.97	10
Chloroform	624	1	51-138	≤25	0.90	5.0
	624(low level)	1	51-138	≤25	0.14	1.0
Chloromethane	624	1	D-273	≤40	2.1	10
	624(low level)	1	D-273	≤40	0.30	1.0
Dibromochloromethane	624	1	53-149	≤25	0.51	5.0
	624(low level)	1	53-149	≤25	0.17	1.0
1,2-Dichlorobenzene	624	1	18-190	≤25	0.44	5.0
	624(low level)	1	18-190	≤25	0.080	1.0
1,3-Dichlorobenzene	624	1	59-156	≤25	0.64	5.0
	624(low level)	1	59-156	≤25	0.11	1.0
1,4-Dichlorobenzene	624	1	18-190	≤25	0.52	5.0
	624(low level)	1	18-190	≤25	0.14	1.0
1,1-Dichloroethane	624	1	59-155	≤40	0.52	5.0
	624(low level)	1	59-155	≤40	0.38	1.0
1,2-Dichloroethane	624	1	49-155	≤26	0.57	5.0
	624(low level)	1	49-155	≤26	0.46	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
1,1-Dichloroethene	624	1	D-234	≤25	0.45	5.0
	624(low level)	1	D-234	≤25	0.19	1.0
trans-1,2-Dichloroethene	624	1	54-156	≤40	0.44	5.0
	624(low level)	1	54-156	≤40	0.19	1.0
1,2-Dichloropropane	624	1	D-210	≤25	0.52	5.0
	624(low level)	1	D-210	≤25	0.090	1.0
cis-1,3-Dichloropropene	624	1	D-227	≤25	0.47	5.0
	624(low level)	1	D-227	≤25	0.17	1.0
trans-1,3-Dichloropropene	624	1	17-183	≤25	0.38	5.0
	624(low level)	1	17-183	≤18	0.49	1.0
Ethylbenzene	624	1	37-162	≤25	0.83	5.0
	624(low level)	1	37-162	≤25	0.11	1.0
Methylene chloride	624	1	D-221	≤37	0.31	5.0
(Dichloromethane)	624(low level)	1	D-221	≤37	0.25	5.0
1,1,2,2-Tetrachloroethane	624	1	46-157	≤25	0.75	5.0
	624(low level)	1	46-157	≤25	0.13	1.0
Tetrachloroethene	624	1	64-148	≤25	1.6	5.0
	624(low level)	1	64-148	≤25	0.38	1.0
Toluene	624	1	47-150	≤25	0.51	5.0
	624(low level)	1	47-150	≤25	0.28	1.0
1,1,1-Trichloroethane	624	1	52-162	≤25	0.46	5.0
	624(low level)	1	52-162	≤25	0.14	1.0
1,1,2-Trichloroethane	624	1	52-150	≤25	0.47	5.0
	624(low level)	1	52-150	≤25	0.15	1.0
Trichloroethene	624	1	71-157	≤25	0.28	5.0
	624(low level)	1	71-157	≤25	0.17	1.0
Trichlorofluoromethane	624	1	17-181	≤65	0.98	5.0
	624(low level)	1	17-181	≤65	0.49	1.0
Vinyl chloride	624	1	D-251	≤40	0.50	10
	624(low level)	1	D-251	≤33	0.28	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Surrogate - Toluene-d8	624	1	77-122	NA	NA	NA
	624(low level)	1	77-122	NA	NA	NA
Surrogate - p-Bromofluorobenzene	624	1	74-126	NA	NA	NA
	624(low level)	1	74-126	NA	NA	NA
Surrogate -Dibromofluoromethane	624	1	70-130	NA	NA	NA
	624(low level)	1	70-130	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	624	1	70-130	NA	NA	NA
	624(low level)	1	70-130	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	624	1	70-130	NA	NA	NA
	624(low level)	1	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Acenaphthene	625	1	47-145	≤30	0.25	10
Acenaphthylene	625	1	33-145	≤38	0.33	10
Aldrin	625	1	D-166	≤25	0.34	10
Anthracene	625	1	27-133	≤27	0.33	10
Benzidine	625	1	D-200	≤100	5.6	80
Benzo(a)anthracene	625	1	33-143	≤32	0.30	10
Benzo(b)fluoranthene	625	1	24-159	≤31	0.28	10
Benzo(k)fluoranthene	625	1	11-162	≤36	0.72	10
Benzo(g,h,i)perylene	625	1	D-219	≤32	0.68	10
Benzo(a)pyrene	625	1	17-163	≤39	0.41	10
alpha-BHC	625	1	10-150	≤50	0.37	10
beta-BHC	625	1	24-149	≤25	0.80	10
delta-BHC	625	1	D-110	≤25	1.7	10
gamma-BHC	625	1	10-150	≤50	0.30	10
Bis(2-chloroethoxy) methane	625	1	33-184	≤35	0.26	10
Bis(2-chloroethyl) ether	625	1	12-158	≤28	0.44	10
Bis(2-chloroisopropyl) ether (2,2-Oxybis(1-chloropropane)	625	1	36-166	≤25	0.23	10
Bis(2-ethylhexyl) phthalate	625	1	8-158	≤39	0.48	10
4-Bromophenyl phenyl ether	625	1	53-127	≤32	0.35	10
Butyl benzyl phthalate	625	1	D-152	≤30	0.41	10
Chlordimeform (Galecron)	625	1	10-150	≤50	13	50
5-Chloroaminotoluene	625	1	10-150	≤50	7.7	10
4-Chloro-3-methylphenol	625	1	22-147	≤29	0.33	10
2-Chloronaphthalene	625	1	60-118	≤25	0.39	10
2-Chlorophenol	625	1	23-134	≤35	0.24	10
4-Chlorophenylphenyl ether	625	1	25-158	≤54	0.66	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Chrysene	625	1	17-168	≤26	0.44	10
1,8-Cineole	625	1	50-101	≤25	0.80	10
p-Cymene	625	1	52-110	≤25	1.6	10
4,4'-DDD	625	1	D-145	≤27	0.35	10
4,4'-DDE	625	1	4-136	≤26	0.30	10
4,4'-DDT	625	1	D-203	≤39	0.40	10
Dibenz(a,h)anthracene	625	1	D-227	≤37	0.80	10
Di-n-butyl phthalate	625	1	1-118	≤25	0.26	10
1,2-Dichlorobenzene	625	1	32-129	≤41	0.31	10
1,3-Dichlorobenzene	625	1	D-172	≤52	0.32	10
1,4-Dichlorobenzene	625	1	20-124	≤43	0.29	10
3,3'-Dichlorobenzidine	625	1	D-262	≤193	4.4	20
2,4-Dichlorophenol	625	1	39-135	≤25	0.66	10
Dieldrin	625	1	29-136	≤25	0.41	10
Diethyl phthalate	625	1	D-114	≤32	0.47	10
2,4-Dimethylphenol	625	1	32-119	≤44	0.39	10
Dimethylphthalate	625	1	D-112	≤25	0.39	10
4,6-Dinitro-2-methylphenol	625	1	D-181	≤39	5.0	50
2,4-Dinitrophenol	625	1	D-191	≤94	8.9	50
2,4-Dinitrotoluene	625	1	39-139	≤54	0.41	10
2,6-Dinitrotoluene	625	1	50-158	≤37	0.34	10
Di-n-octyl phthalate	625	1	4-146	≤32	0.35	10
Endosulfan I	625	1	10-150	≤50	1.8	20
Endosulfan II	625	1	10-150	≤50	1.3	20
Endosulfan sulfate	625	1	D-107	≤25	0.75	20
Endrin	625	1	10-150	≤50	2.3	20
Endrin aldehyde	625	1	D-209	≤59	18	50
Fluoranthene	625	1	26-137	≤27	0.33	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Fluorene	625	1	59-121	≤25	0.38	10
Heptachlor	625	1	D-192	≤36	0.42	20
Heptachlor epoxide	625	1	26-155	≤25	0.58	20
Hexachlorobenzene	625	1	D-152	≤25	0.19	10
Hexachlorobutadiene	625	1	24-116	≤28	0.35	10
Hexachlorocyclopentadiene	625	1	D-200	≤50	2.4	10
Hexachloroethane	625	1	40-113	≤55	0.32	10
Indeno(1,2,3-cd)pyrene	625	1	D-171	≤37	0.56	10
Isophorone	625	1	21-196	≤25	0.37	10
d-Limonene	625	1	36-74	≤25	1.4	10
2,4(8)-p-Menthadiene	625	1	22-85	≤42	1.6	10
Naphthalene	625	1	21-133	≤36	0.36	10
Nitrobenzene	625	1	35-180	≤33	0.31	10
2-Nitrophenol	625	1	29-182	≤28	0.36	10
4-Nitrophenol	625	1	D-132	≤39	4.9	50
N-Nitrosodimethylamine	625	1	10-150	≤41	0.49	10
N-Nitrosodiphenylamine/Diphenylamine (requires floracil cleanup to separate N-Nitrosodiphenyamine from Diphenylamine)	625	1	10-150	≤50	0.34	10
N-Nitrosodi-n-propylamine	625	1	D-230	≤39	0.29	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Pentachlorophenol	625	1	14-176	≤37	4.0	50
beta-Phellandrene	625	1	33-65	≤25	1.9	10
Phenanthrene	625	1	54-120	≤27	0.33	10
Phenol	625	1	5-112	≤37	0.28	10
cis-Pinane	625	1	50-103	≤29	2.9	10
alpha-Pinene	625	1	12-105	≤25	1.3	100
beta-Pinene	625	1	DL-155	≤56	2.0	100
Pyrene	625	1	52-115	≤36	0.53	10
Pyridine	625	1	10-150	≤50	0.93	50
alpha-Terpinene	625	1	DL-94	≤96	1.2	10
Terpineol	625	1	50-150	≤25	0.82	10
Terpinolene	625	1	28-81	≤45	1.7	10
o-Toluidine	625	1	10-150	≤50	0.53	10
1,2,4-Trichlorobenzene	625	1	44-142	≤28	0.36	10
2,4,6-Trichlorophenol	625	1	37-144	≤30	0.35	10
Surrogate - Nitrobenzene-d5	625	1	34-130	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	625	1	36-124	NA	NA	NA
Surrogate - Phenol-d5	625	1	25-128	NA	NA	NA
Surrogate – 2-Fluorophenol	625	1	29-121	NA	NA	NA
Surrogate - 2,4,6-Tribromophenol	625	1	29-143	NA	NA	NA
Surrogate - Terpehnyl-d14	625	1	14-148	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Benfluralin	627	9	40-140	≤40	0.0025	0.010
Ethalfluralin	627	9	40-140	≤50	0.50	2.0
Isopropalin	627	9	48-140	≤50	0.025	0.10
Profluralin	627	9	55-140	≤50	0.050	0.20
Trifluralin (MS)	627	9	17-140	≤50	0.0025	0.010
Surrogate - 2,4,5,6-Tetrachloro-m-xylene	627	9	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Cyanizine	629	25	27-133	≤40	0.12	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Amobam	630	63	70-130	≤20	♦	♦
Ferbam	630	63	70-130	≤20	♦	♦
Mancozeb	630	63	70-130	≤20	♦	♦
Maneb	630	63	70-130	≤20	♦	♦
Metham	630	63	70-130	≤20	♦	♦
Nabam	630	63	70-130	≤20	♦	♦
Polyram	630	63	70-130	≤20	♦	♦
Zineb	630	63	70-130	≤20	♦	♦
Ziram	630	63	70-130	≤20	1.5	20

♦ All compounds reported as Ziram

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Benomyl (as Carbendazim)	631	55	13-137	≤50	0.046	0.50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Aminocarb	632	13	60-125	≤30	0.25	1.0
Barban	632	13	55-125	≤30	0.21	1.0
Bromacil	632	13	52-125	≤30	0.19	2.0
Carbaryl (MS)	632	13	55-125	≤30	0.19	2.0
Carbofuran	632	13	55-125	≤30	0.45	10
Chlorphropham	632	13	55-125	≤30	0.21	1.0
Diuron (MS)	632	13	55-125	≤30	0.061	0.20
Fenuron	632	13	60-125	≤30	0.067	5.0
Fluomethuron	632	13	59-125	≤40	0.095	1.0
Linuron	632	13	55-125	≤30	0.058	0.50
Methomyl	632	13	52-132	≤30	0.30	5.0
Methiocarb	632	13	51-137	≤30	0.37	5.0
Monuron	632	13	56-132	≤30	0.089	1.0
Neburon	632	13	54-126	≤30	0.029	1.0
Oxamyl	632	13	57-125	≤30	0.24	10
Propham	632	13	50-125	≤30	0.21	1.0
Propoxur	632	13	56-125	≤30	2.1	5.0
Siduron	632	13	55-125	≤30	0.25	1.0
Swep	632	13	58-125	≤30	0.076	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Bromacil	633	41	52-125	≤30	0.50	2.0
DEET	633	41	52-125	≤30	1.2	5.0
Hexazinone	633	41	52-125	≤30	0.12	0.50
Metribuzin	633	41	50-125	≤30	0.25	1.0
Terbacil	633	41	50-130	≤30	1.2	5.0
Triadimefon	633	41	48-125	≤30	0.25	1.0
Tricyclazole	633	41	53-125	≤30	1.2	5.0
Surrogate - Triphenylphosphate	633	41	16-164	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Butylate (MS)	634	15	38-145	≤76	0.28	2.0
Cycloate	634	15	46-159	≤47	0.24	2.0
EPTC	634	15	46-154	≤55	0.42	2.0
Molinate (MS)	634	15	37-127	≤74	0.21	2.0
Pebulate	634	15	22-172	≤50	0.23	2.0
Vernolate	634	15	39-147	≤45	0.19	2.0
Surrogate - Triphenylphosphate	634	15	16-164	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS						
PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Alachlor (MS)	645	28	45-140	≤30	0.25	1.0
Butachlor	645	28	50-124	≤40	0.25	1.0
Diphenamid	645	28	57-119	≤40	0.25	1.0
Fluridone	645	28	45-154	≤40	0.25	1.0
Lethane	645	28	33-153	≤50	0.25	1.0
Norflurazon	645	28	48-110	≤40	0.25	1.0
Surrogate - Triphenylphosphate	645	28	16-164	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS						
PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL (ug/L)	RLA (ug/L)
Monochlorobiphenyls	680	93	30-130	≤50	0.044	0.10
Dichlorobiphenyls	680	93	30-130	≤50	0.035	0.10
Trichlorobiphenyls	680	93	30-130	≤50	0.035	0.10
Tetrachlorobiphenyls	680	93	40-140	≤50	0.053	0.20
Pentachlorobiphenyls	680	93	40-140	≤50	0.029	0.20
Hexachlorobiphenyls	680	93	40-140	≤50	0.037	0.20
Heptachlorobiphenyls	680	93	40-140	≤50	0.042	0.30
Octachlorobiphenyls	680	93	40-140	≤50	0.064	0.30
Nonachlorobiphenyls	680	93	30-130	≤50	0.11	0.50
Decachlorobiphenyl	680	93	30-130	≤50	0.11	0.50
Surrogate-Decachlorobiphenyl-13C10	680	93	30-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Pulp and Paper Samples						
Chloroform	1624	1	40-150	≤25	0.91	5.0
Internal Standard- Chloroform-13C	1624	1	18-172	NA	NA	NA
Chloroform	624	1	51-138	≤25	0.90	5.0
Surrogate - Toluene-d8	624	1	77-122	NA	NA	NA
Surrogate - p-Bromofluorobenzene	624	1	74-126	NA	NA	NA
Surrogate --Dibromofluoromethane	624	1	D-130(1)	NA	NA	NA

(1) D = detected. This surrogate shows very low bias in alkali samples.

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
2,4,6-Trichlorophenol	1653	94	72-146	≤37	0.37	2.5
2,4,5-trichlorophenol	1653	94	82-128	≤33	0.070	2.5
2,3,4,6-Tetrachlorophenol	1653	94	82-132	≤26	0.57	2.5
3,4,6-Trichloroguaicol	1653	94	74-140	≤33	0.26	2.5
3,4,5-Trichloroguaicol	1653	94	80-134	≤27	0.31	2.5
4,5,6-Trichloroguaicol	1653	94	88-116	≤25	0.16	2.5
3,4,6-Trichloroatechol	1653	94	64-149	≤43	0.80	5.0
Pentachlorophenol	1653	94	84-120	≤25	0.57	5.0
3,4,5-Trichloroatechol	1653	94	72-128	≤28	0.92	5.0
Tetrachloroguaicol	1653	94	81-126	≤25	0.85	5.0
Trichlorosyringol	1653	94	66-174	≤27	0.52	2.5
Tetrachlorocatechol	1653	94	81-132	≤32	0.13	5.0
Internal Standard- 3,4,5-Trichlorophenol	1653	94	56-116 (1) 24-167 (2)	NA	NA	NA
Internal Standard- 4,5,6-Trichloroguaaiacol-13C6	1653	94	48-131 (1) 51-139 (2)	NA	NA	NA
Internal Standard- Pentachlorophenol-13C6	1653	94	8-143 (1) 27-167 (2)	NA	NA	NA
Internal Standard- Tetrachloroguaiacol-13C6	1653	94	35-120 (1) 27-161 (2)	NA	NA	NA
Internal Standard- Tetrachlorocatechol-13C6	1653	94	14-118 (1) 0-184 (2)	NA	NA	NA

(1) Recovery limits for the internal standards with ascorbic acid ; (2) Recovery limits for the internal standards without ascorbic acid.

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RLA (ug/L)
Chloropicrin	8011	2	60-140	≤40	0.0025	0.010
1,2-Dibromoethane (EDB)	8011	2	60-140	≤40	0.0061	0.020
1,2-Dibromo 3-chloropropane (DBCP)	8011	2	60-140	≤40	0.0041	0.020
1,1-Dichloropropane	8011	2	60-140	≤40	0.50	2.0
1,3-Dichloropropene	8011	2	60-140	≤40	0.25	1.0
Methyl isothiocyanate	8011**V	2	60-140	≤40	5.0	20

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Acetone (MS)	8015(5030)	2	40-130	≤30	6.8	25
2-Butanone (MEK) (MS)	8015(5030)	2	60-130	≤40	8.8	25
Diethyl ether (Ethyl ether)	8015(5030)	2	10-130	≤50	2.0	25
Heptane	8015(5030)	2	70-130	≤30	0.50	1.0
Hexane	8015(5030)	2	70-130	≤30	0.50	1.0
2-Hexanone	8015(5030)	2	50-150	≤50	1.6	25
4-Methyl-2-pentanone (MIBK) (MS)	8015(5030)	2	65-125	≤40	4.0	25
Methyl t-butyl ether (MTBE)	8015(5030)	2	40-140	≤50	0.59	10
Gasoline	GRO(TENNESSEE)	70	50-100	≤20	15	36
	GRO(8015 modified volatiles)	2	50-150	≤40	17	50
	8015(modified volatiles) (5030)	2/12	50-150	≤40	17	50
Volatile Petroleum Hydrocarbons	TPH	107	70-130	≤25	9.7	100
(VPH by Massachusetts Method)	C5-C8 Aliphatic Hydrocarbons	107	70-130	≤25	8.6	40
	C9-C12 Aliphatic Hydrocarbons	107	70-130	≤25	3.5	10
	C9-C10 Aromatic Hydrocarbons	107	70-130	≤25	0.70	10
Lacolene	8015 (modified volatiles) (5030)	2/12	40-140	≤40	25	50
Surrogate - a,a,a-Trifluorotoluene	GRO (TENNESSEE)	70	50-150	NA	NA	NA
	8015 (5030)	2/12	23-164	NA	NA	NA
	Ma VPH	107	60-140	NA	NA	NA
Surrogate - 2,5-Dibromotoluene	Ma VPH	107	60-140	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Petroleum hydrocarbons	FL-PRO	83	41-101	≤20	100	300
	8015 (modified extractable) (3510/3520)	2	30-139	≤40	100	300
Extractable Petroleum Hydrocarbons	TPH	106	40-140	≤25	22	100
(EPH by Massachusetts's Method)	C9-C18 Aliphatic Hydrocarbons	106	40-140	≤25	9.3	100
	C19-C36 Aliphatic Hydrocarbons	106	40-140	≤25	14	100
	C11-C22 Aromatic hydrocarbons	106	40-140	≤25	22	100
Texas TPH	TPH	114	70-130	≤30	1900	5000
	C6-C10 Hydrocarbons	114	70-130	≤30	3700	5000
	C10-C28 Hydrocarbons	114	70-130	≤30	2300	5000
Diesel	DRO	69/2	40-140	≤40	75	100
	8015 (modified extractable) (3510/3520)	12/2	10-114	≤40	75	300
Heavy oil	8015 (modified extractable) (3510/3520)	12/2	30-165	≤40	510	3000
Kerosene	8015 (modified extractable) (3510/3520)	12/2	10-133	≤60	36	300
Mineral-Spirits	8015 (modified extractable) (3510/3520)	2	42-145	≤46	88	300
Surrogate - 2-Fluorobiphenyl	8015 (modified extractable) (3510/3520)	2/12	10-130	NA	NA	NA
Surrogate - o-Terphenyl	DRO	69/12	38-156	NA	NA	NA
	8015 (modified extractable) (3510/3520)	2	38-156	NA	NA	NA
	FL-PRO	83	38-156	NA	NA	NA
	EPH	106	40-140	NA	NA	NA
Surrogate-Nonatricontane (C39)	FL-PRO	83	24-137	NA	NA	NA
Surrogate - Chloro-octadecane	EPH	106	40-140	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
ACETATES						
n-Butyl acetate	8015 (DAI)	2	50-150	≤50	350	5000
Sec-Butyl acetate	8015 (DAI)	2	50-150	≤50	290	5000
Cellosolve acetate	8015 (DAI)	2	50-150	≤50	430	5000
Ethyl acetate (MS)	8015 (DAI)	2	43-160	≤50	630	5000
Isoamyl acetate	8015 (DAI)	2	50-150	≤50	490	5000
Isobutyl acetate	8015 (DAI)	2	67-166	≤50	930	5000
Methyl acetate	8015 (DAI)	2	46-170	≤50	910	5000
Isopropyl acetate	8015 (DAI)	2	67-166	≤50	460	5000
Phenyl mercuric acetate	8015 (DAI)	2	30-130	≤50	10000	20000
n-Propyl acetate (MS)	8015 (DAI)	2	24-158	≤50	460	5000
ALCOHOLS						
Tert-Amyl alcohol	8015 (DAI)	2	49-181	≤50	530	1000
Isobutanol	8015 (DAI)	2	50-150	≤50	710	1000
n-Butanol	8015 (DAI)	2	50-150	≤50	700	1000
Sec-Butanol	8015 (DAI)	2	37-182	≤50	390	1000
Tert-Butanol	8015 (DAI)	2	67-183	≤50	480	1000
Diacetone alcohol	8015 (DAI)	2	50-150	≤50	680	5000
Ethanol (MS)	8015 (DAI)	2	60-129	≤50	840	1000
Methanol (MS)	8015 (DAI)	2	61-128	≤50	800	1000
n-Propanol	8015 (DAI)	2	50-150	≤50	680	1000
Isopropanol (MS)	8015 (DAI)	2	61-148	≤50	510	1000
CELLOSOLVES						
Butyl cellosolve	8015 (DAI)	2	50-150	≤50	390	5000
Ethyl cellosolve (2-Ethoxyethanol)	8015 (DAI)	2	20-180	≤50	ND	20000

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
GLYCOLS						
Diethylene glycol	8015 (DAI)	2	45-112	≤50	1200	5000
Ethylene glycol (MS)	8015 (DAI)	2	35-132	≤50	890	5000
Propylene glycol (MS)	8015 (DAI)	2	60-126	≤50	600	5000
Tetraethylene glycol	8015 (DAI)	2	50-150	≤50	1200	10000
Triethylene glycol	8015 (DAI)	2	50-150	≤50	2200	5000
MICELLANEOUS SOLVENTS						
Cyclohexanone	8015 (DAI)	2	50-150	≤50	670	5000
1,4-Dioxane	8015 (DAI)	2	50-150	≤50	320	5000
Mesityl oxide	8015 (DAI)	2	50-150	≤50	260	5000
2-Nitropropane	8015 (DAI)	2	50-150	≤50	520	5000
Tetrahydrofuran (MS)	8015 (DAI)	2	67-146	≤50	290	5000

- DAI = Direct Aqueous Injection

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Acetaldehyde (MS)	8015 (DAI*)(NCASI)	2/108	70-130	≤30	260	500
Methanol (MS)	8015 (DAI*)(NCASI)	2/108	70-130	≤30	320	500
Acrolein	8015 (DAI*)(NCASI)	2/108	70-130	≤30	60	500
Propionaldehyde	8015 (DAI*)(NCASI)	2/108	70-130	≤30	110	500
Acetone	8015 (DAI*)(NCASI)	2/108	70-130	≤30	190	500
Methyl ethyl ketone (MEK) (MS)	8015 (DAI*)(NCASI)	2/108	70-130	≤30	120	500
Methyl iso-butyl ketone (MIBK)	8015 (DAI*)(NCASI)	2/108	70-130	≤30	160	500
Phenol	8015 (DAI*)(NCASI)	2/108	70-130	≤30	400	500

- DAI = Direct Aqueous Injection

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Benzene (MS)	8021(5030)	2	48-152	≤24	0.10	1.0
Bromobenzene	8021(5030)	2	46-195	≤56	0.23	5.0
Bromochloromethane	8021(5030)	2	34-128	≤52	0.15	1.0
Bromodichloromethane	8021(5030)	2	68-136	≤54	0.070	1.0
Bromoform	8021(5030)	2	74-133	≤40	0.27	5.0
Bromomethane	8021(5030)	2	38-179	≤28	0.090	1.0
n-Butylbenzene	8021(5030)	2	50-150	≤25	0.11	1.0
sec-Butylbenzene	8021(5030)	2	50-150	≤25	0.55	1.0
tert-Butylbenzene	8021(5030)	2	49-188	≤48	0.18	1.0
Carbon tetrachloride	8021(5030)	2	74-128	≤29	0.18	1.0
Chlorobenzene (MS)	8021(5030)	2	56-134	≤37	0.11	1.0
Chloroethane	8021(5030)	2	38-170	≤52	0.24	1.0
2-Chloroethylvinyl ether	8021(5030)	2	D-130	≤56	2.5	10
Chloroform	8021(5030)	2	48-131	≤38	0.11	1.0
Chloromethane	8021(5030)	2	21-136	≤56	0.14	1.0
2-Chlorotoluene	8021(5030)	2	50-150	≤30	0.35	5.0
4-Chlorotoluene	8021(5030)	2	50-150	≤30	0.17	5.0
Dibromochloromethane	8021(5030)	2	52-134	≤31	0.10	1.0
1,2-Dibromo-3-chloropropane (DBCP)	8021(5030)	2	24-145	≤56	1.6	5.0
1,2-Dibromoethane (EDB)	8021(5030)	2	34-168	≤77	0.15	2.0
Dibromomethane	8021(5030)	2	41-147	≤59	0.32	2.0
1,2-Dichlorobenzene	8021(5030)	2	70-130	≤19	0.21	1.0
1,3-Dichlorobenzene	8021(5030)	2	35-137	≤17	0.19	1.0
1,4-Dichlorobenzene	8021(5030)	2	70-130	≤32	0.16	1.0
Dichlorodifluoromethane	8021(5030)	2	57-124	≤50	0.23	1.0
1,1-Dichloroethane	8021(5030)	2	54-129	≤22	0.13	1.0
1,2-Dichloroethane	8021(5030)	2	51-131	≤32	0.12	1.0
1,1-Dichloroethene (MS)	8021(5030)	2	37-163	≤40	0.13	1.0
cis-1,2-Dichloroethene	8021(5030)	2	62-120	≤24	0.10	1.0
trans-1,2-Dichloroethene	8021(5030)	2	61-130	≤26	0.12	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
1,2-Dichloropropane	8021(5030)	2	46-134	≤30	0.14	1.0
1,3-Dichloropropane	8021(5030)	2	53-150	≤57	0.16	1.0
2,2-Dichloropropane	8021(5030)	2	40-138	≤39	0.39	1.0
1,1-Dichloropropene	8021(5030)	2	42-141	≤50	0.10	1.0
cis-1,3-Dichloropropene	8021(5030)	2	51-131	≤34	0.26	1.0
trans-1,3-Dichloropropene	8021(5030)	2	54-137	≤18	0.14	1.0
Ethylbenzene	8021(5030)	2	54-125	≤22	0.14	1.0
Hexachlorobutadiene	8021(5030)	2	52-145	≤41	0.62	1.0
Isopropylbenzene	8021(5030)	2	50-150	≤27	0.13	1.0
p-Isopropyltoluene	8021(5030)	2	50-150	≤25	0.15	1.0
Methylene chloride (Dichloromethane)	8021(5030)	2	39-164	≤23	0.30	5.0
Methyl t-butyl ether (MTBE)	8021(5030)	2	40-140	≤50	0.59	10
Naphthalene	8021(5030)	2	70-130	≤30	0.68	1.0
n-Propylbenzene	8021(5030)	2	50-150	≤25	0.31	1.0
Styrene	8021(5030)	2	70-130	≤30	0.17	1.0
1,1,1,2-Tetrachloroethane	8021(5030)	2	62-141	≤41	0.46	1.0
1,1,2,2-Tetrachloroethane	8021(5030)	2	71-132	≤48	0.070	1.0
Tetrachloroethene (Tetrachloroethylene)	8021(5030)	2	74-127	≤30	0.12	1.0
Toluene (MS)	8021(5030)	2	62-133	≤27	0.13	1.0
1,2,3-Trichlorobenzene	8021(5030)	2	56-108	≤26	0.55	1.0
1,2,4-Trichlorobenzene	8021(5030)	2	44-139	≤53	0.26	1.0
1,1,1-Trichloroethane	8021(5030)	2	71-134	≤36	0.10	1.0
1,1,2-Trichloroethane	8021(5030)	2	52-124	≤20	0.070	1.0
Trichloroethene (Trichloroethylene) (MS)	8021(5030)	2	49-138	≤49	0.10	1.0
Trichlorofluoromethane	8021(5030)	2	49-140	≤37	0.18	1.0
1,2,3-Trichloropropane	8021(5030)	2	61-148	≤48	0.23	2.0
1,2,4-Trimethylbenzene	8021(5030)	2	32-132	≤44	0.19	1.0
1,3,5-Trimethylbenzene	8021(5030)	2	50-150	≤25	0.13	1.0
Vinyl Chloride	8021(5030)	2	59-136	≤47	0.22	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
o-Xylene	8021(5030)	2	54-128	≤21	0.11	1.0
m&p-Xylene	8021(5030)	2	54-125	≤30	0.27	1.0
Total Xylenes	8021(5030)	2	54-125	≤30	0.27	2.0
Surrogate* - 2-Bromo-1-chloropropane	8021(5030)	2	70-130	NA	NA	NA
Surrogate *- Fluorobenzene	8021(5030)	2	70-130	NA	NA	NA
Surrogate* - 1-Bromo-3-chloropropane	8021(5030)	2	70-130	NA	NA	NA
Surrogate- Bromochloromethane	8021(5030)	2	70-130	NA	NA	NA
Surrogate*- a,a,a-Trifluorotoluene	8021(5030)	2	70-130	NA	NA	NA
Surrogate- 1,4-Dichlorobutane	8021(5030)	2	70-130	NA	NA	NA
Surrogate- Bromochlorobenzene	8021(5030)	2	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
2-Chlorophenol (MS)	8041(3510/3520)	2	10-164	≤28	1.2	10
4-Chloro-3-methylphenol (MS)	8041(3510/3520)	2	10-165	≤33	1.2	10
2,4-Dichlorophenol	8041(3510/3520)	2	56-106	≤21	1.3	10
2,6-Dichlorophenol	8041(3510/3520)	2	68-110	≤23	1.4	10
2,4-Dimethylphenol	8041(3510/3520)	2	56-111	≤30	1.3	10
2,4-Dinitrophenol	8041(3510/3520)	2	28-119	≤36	1.1	50
2-Methyl-4,6-dinitrophenol	8041(3510/3520)	2	28-154	≤33	1.1	50
3- and 4-Methylphenol (m & p cresol)	8041(3510/3520)	2	10-150	≤50	3.3	10
2-Methylphenol (o-cresol)	8041(3510/3520)	2	10-150	≤50	1.8	10
Cresols (total)	8041(3510/3520)	2	NA	NA	5.1	10
2-Nitrophenol	8041(3510/3520)	2	49-111	≤26	1.0	10
4-Nitrophenol (MS)	8041(3510/3520)	2	10-171	≤33	1.2	50
Pentachlorophenol (MS)	8041(3510/3520)	2	10-216	≤28	1.3	50
Phenol (MS)	8041(3510/3520)	2	10-147	≤56	1.0	10
2,3,4,5-Tetrachlorophenol	8041(3510/3520)	2	50-150	≤40	2.5	20
2,3,4,6-Tetrachlorophenol	8041(3510/3520)	2	50-150	≤40	3.1	20
Tetrachlorophenols (2,3,4,5 & 2,3,4,6)	8041(3510/3520)	2	50-150	≤40	5.6	20
2,4,5-Trichlorophenol	8041(3510/3520)	2	53-119	≤40	4.4	10
2,4,6-Trichlorophenol	8041(3510/3520)	2	56-114	≤30	1.9	10
Trichlorophenols (2,4,5 & 2,4,6)	8041(3510/3520)	2	53-119	≤40	6.3	10
Surrogate - 2,4,6-Tribromophenol	8041(3510/3520)	2	14-144	NA	NA	NA
Surrogate - 2-Fluorophenol	8041(3510/3520)	2	29-121	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RLA (ug/L)
Bis(2-ethylhexyl) phthalate (MS)	8061(3510/3520)	2	28-139	≤82	1.2	10
Butyl benzyl phthalate (MS)	8061(3510/3520)	2	42-152	≤73	1.3	10
Diethyl phthalate (MS)	8061(3510/3520)	2	43-150	≤47	1.8	10
Dimethyl phthalate (MS)	8061(3510/3520)	2	40-148	≤63	1.8	10
Di-n-butyl phthalate (MS)	8061(3510/3520)	2	41-154	≤46	1.8	10
Di-n-octyl phthalate (MS)	8061(3510/3520)	2	29-136	≤52	2.7	10
Surrogate - 2-Fluorobiphenyl	8061(3510/3520)-FID	2	25-109	NA	NA	NA
Surrogate - 2,3,4,6-Tetrachloro-m-xylene (TCMX)	8061(3510/3520)-EC	2	30-150	NA	NA	NA
Surrogate – Decachlorobiphenyl (DCB)	8061(3510/3520)-EC	2	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (%Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
N-Nitrosodimethylamine	8070(3510/3520)	2	13-109	≤38	1.7	2.0
N-Nitrosodi-n-propylamine	8070(3510/3520)	2	45-146	≤75	1.8	2.0
1,3-Dinitrobenzene	8070(3520)	2	67-115	≤30	0.96	3.0
N-Nitrosodiphenylamine	8070(3510/3520)	2	10-139	≤67	2.1	3.0
p-Dimethylaminoazobenzene	8070(3520)	2	69-140	≤48	0.94	3.0
Surrogate - Tripehnylphosphate	8070(3510/3520)	2	16-164	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RLA (ug/L)
Alachlor	8081(3510/3520)	2	62-128	≤30	0.41	2.0
Aldrin (MS)	8081(3510/3520)	2	38-129	≤25	0.0099	0.050
Benfluralin	8081(3520)	2	40-140	≤40	0.0025	0.020
alpha BHC	8081(3510/3520)	2	46-131	≤30	0.0079	0.050
beta BHC	8081(3510/3520)	2	36-153	≤35	0.0074	0.050
delta BHC	8081(3510/3520)	2	53-137	≤41	0.012	0.050
gamma BHC (Lindane) (MS)	8081(3510/3520)	2	40-139	≤26	0.0074	0.050
Captafol	8081(3510/3520)	2	20-180	≤50	0.025	0.10
alpha Chlordane	8081(3510/3520)	2	55-125	≤17	0.0076	0.050
gamma Chlordane	8081(3510/3520)	2	54-128	≤18	0.0074	0.050
technical Chlordane	8081(3510/3520)	2	54-140	≤30	0.043	0.50
Chlorobenzilate	8081(3510/3520)	2	50-150	≤40	0.078	0.50
Chloroneb	8081(3510/3520)	2	49-125	≤30	0.091	0.40
Chloropropylate	8081(3510/3520)	2	51-125	≤30	0.17	0.50
Chlorothalonil	8081(3510/3520)	2	55-125	≤30	0.050	0.20
Dacthal (DCPA)	8081(3510/3520)	2	50-150	≤25	0.014	0.020
4,4'-DDD	8081(3510/3520)	2	32-155	≤39	0.018	0.10
4,4'-DDE	8081(3510/3520)	2	48-145	≤18	0.014	0.10
4,4'-DDT (MS)	8081(3510/3520)	2	50-147	≤27	0.017	0.10
Dicofol (Kelthane)	8081(3510/3520)	2	55-115	≤40	0.0067	0.050
Dieldrin (MS)	8081(3510/3520)	2	34-150	≤42	0.012	0.10
Endosulfan I	8081(3510/3520)	2	34-161	≤24	0.0094	0.050
Endosulfan II	8081(3510/3520)	2	40-162	≤22	0.018	0.10
Endosulfan sulfate	8081(3510/3520)	2	28-156	≤28	0.020	0.10
Endrin (MS)	8081(3510/3520)	2	41-158	≤25	0.014	0.10
Endrin aldehyde	8081(3510/3520)	2	20-146	≤34	0.021	0.10
Endrin ketone	8081(3510/3520)	2	42-122	≤25	0.020	0.10
Etradiazole	8081(3510/3520)	2	60-125	≤30	0.0018	0.020
Heptachlor (MS)	8081(3510/3520)	2	37-148	≤26	0.0062	0.050
Heptachlor epoxide	8081(3510/3520)	2	43-141	≤31	0.0069	0.050

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RLA (ug/L)
Hexachlorobenzene	8081(3510/3520)	2	25-112	≤33	0.0070	0.050
Isodrin	8081(3510/3520)	2	55-110	≤40	0.019	0.050
Kepone	8081(3510/3520)	2	10-150	≤50	0.038	1.0
Methoxychlor	8081(3510/3520)	2/26	60-155	≤43	0.038	0.50
Mirex	8081(3510/3520)	2	52-112	≤37	0.046	0.50
PCNB	8081(3510/3520)	2	60-125	≤30	0.0033	0.60
Pendimethalin	8081(3510/3520)	2	50-146	≤25	0.0021	0.050
Permethrin	8081(3510/3520)	2	50-130	≤40	0.12	1.0
Propachlor	8081(3510/3520)	2	51-125	≤30	0.084	0.50
Toxaphene	8081(3510/3520)	2	12-130	≤30	1.0	5.0
Trifluralin	8081(3510/3520)	2	54-124	≤40	0.0026	0.020
PCB 1016(MS)	8082(3510/3520)	2	45-134	≤34	0.21	1.0
PCB 1221	8082(3510/3520)	2	20-173	≤110	0.36	2.0
PCB 1232	8082(3510/3520)	2	10-228	≤86	0.095	1.0
PCB 1242	8082(3510/3520)	2	37-160	≤74	0.20	1.0
PCB 1248	8082(3510/3520)	2	50-113	≤30	0.13	1.0
PCB 1254	8082(3510/3520)	2	50-122	≤39	0.22	1.0
PCB 1260(MS)	8082(3510/3520)	2	41-144	≤34	0.11	1.0
PCB 1268	8082(3510/3520)	2	40-140	≤40	0.11	1.0
Surrogate – Dibutylchlorendate (DBC)	8081/8082(3510/3520)	2	30-150	NA	NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081/8082 (3510/3520)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	8081/8082 (3510/3520)	2	30-150	NA	NA	NA

NOTE: If only PCBs are requested, PCB1016 and PCB1260 are the matrix spiking compounds.

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
2,4'-Dichlorobiphenyl (8)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',5-Trichlorobiphenyl (18)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,4,4'-Trichlorobiphenyl (28)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',3,5'-Tetrachlorobiphenyl (44)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',5,5'-Tetrachlorobiphenyl (52)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,3',4,4'-Tetrachlorobiphenyl (66)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
3,3',4,4'-Tetrachlorobiphenyl (77)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',4,5,5'-Pentachlorobiphenyl (101)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,3,3',4,4'-Pentachlorobiphenyl (105)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,3',4,4',5-Pentachlorobiphenyl (118)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
3,3',4,4',5-Pentachlorobiphenyl (126)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',3,3',4,4'-Hexachlorobiphenyl (128)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',3,4,4',5'-Hexachlorobiphenyl (138)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',4,4',5,5'-Hexachlorobiphenyl (153)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,3,3',4,4',5-Hexachlorobiphenyl (156)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,3,3',4,4',6-Hexachlorobiphenyl (158)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,3',4,4',5,5'-Hexachlorobiphenyl (167)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
3,3',4,4',5,5'-Hexachlorobiphenyl (169)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',3,3',4,4',5-Heptachlorobiphenyl (170)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',3,4,4',5,5'-Heptachlorobiphenyl (180)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',3,4',5,5',6-Heptachlorobiphenyl (187)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',3,3',4,4',5,6-Octachlorobiphenyl (195)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (206)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
Decachlorobiphenyl (209)	8082(3510/3520)	2	50-150	≤50	0.025	0.10
Surrogate - 2,4,5,6-Tetrachloro-m- xylene (TCMX)	8082(3510/3520)	2	30-150	NA	NA	NA
Surrogate- Octachloronaphthalene	8082(3510/3520)	2	25-150	NA	NA	NA

*These congeners are representative of the 209 individual PCBs that may be determined using Method 8082. The number in parenthesis after the compound name is the PCB congener number. See the previous section for PCBs as Aroclors.

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/L)	RLA (ug/L)
2,4-Dinitrotoluene (MS)	8091(3510/3520)(ECD)	2	10-125	≤40	0.12	0.30
2,6-Dinitrotoluene (MS)	8091(3510/3520)(ECD)	2	10-126	≤40	0.077	0.30
Surrogate - 2,4,5,6-Tetrachloro-m- xylene (TCMX)	8091(3510/3520)(ECD)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	8091(3510/3520)(ECD)	2	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RLA (ug/L)
Anthracene*4	8100(3510/3520)	2	29-164	≤28	1.8	10
Acenaphthene (MS)	8100(3510/3520)	2	27-133	≤25	1.7	10
Acenaphthylene	8100(35100/3520)	2	27-130	≤30	1.9	10
Benzo(a)anthracene*1	8100(3510/3520)	2	35-142	≤38	2.0	10
Benzo(a)pyrene (MS)	8100(3510/3520)	2	30-136	≤40	1.2	10
Benzo(b)fluoranthene*2	8100(3510/3520)	2	32-141	≤51	2.8	10
Benzo(k)fluoranthene*2	8100(3510/3520)	2	32-141	≤51	2.8	10
Benzo(g,h,i)perylene	8100(3510/3520)	2	27-143	≤61	1.6	10
Carbazole	8100(3510/3520)	2	16-140	≤40	1.9	10
Chrysene*1	8100(3510/3520)	2	35-142	≤38	2.0	10
Dibenzo(a,h)anthracene*3	8100(3510/3520)	2	33-154	≤52	1.7	10
Fluoranthene	8100(3510/3520)	2	37-145	≤25	1.7	10
Fluorene (MS)	8100(3510/3520)	2	30-140	≤25	1.7	10
Indeno(1,2,3-cd) pyrene*3	8100(3510/3520)	2	33-154	≤52	2.9	10
1-Methyl naphthalene	8100(3510/3520)	2	10-145	≤50	1.4	10
2-Methyl naphthalene	8100(3510/3520)	3	14-138	≤50	1.2	10
Naphthalene (MS)	8100(3510/3520)	2	16-108	≤28	1.6	10
Phenanthrene*4	8100(3510/3520)	2	33-146	≤28	1.9	10
Pyrene (MS)	8100(3510/3520)	2	36-146	≤28	1.7	10
Surrogate - 2-Fluorobiphenyl	8100(3510/3520)	2	10-130	NA	NA	NA
Surrogate - o-Terphenyl	8100(3510/3520)	2	38-156	NA	NA	NA

*# = Where the number is the same for any 2 compounds, these compounds cannot be routinely resolved chromatographically and are therefore reported as a combined result.

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Bis(2-chloroethoxy)methane	8111(3510/3520)	2	12-128	≤50	1.0	5.0
Bis(2-chloroethyl)ether	8111(3510/3520)	2	11-152	≤50	1.8	20
Bis(2-chloroisopropyl)ether	8111(3510/3520)	2	9-165	≤50	2.5	10
4-Bromophenyl phenyl ether	8111(3510/3520)	2	D-189	≤50	1.3	5.0
4-Chlorophenyl phenyl ether	8111(3510/3520)	2	D-170	≤50	8.8	40
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8111(3510/3520)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	8111(3510/3520)	2	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
2-Chloronaphthalene	8121(3510/3520)	2	9-148	≤50	1.4	10
1,2-Dichlorobenzene	8121(3510/3520)	2	9-160	≤50	0.20	10
1,3-Dichlorobenzene	8121(3510/3520)	2	D-150	≤50	0.24	10
1,4-Dichlorobenzene	8121(3510/3520)	2	13-137	≤50	0.57	10
Hexachlorobenzene	8121(3510/3520)	2	15-159	≤50	0.0032	0.10
Hexachlorobutadiene	8121(3510/3520)	2	D-139	≤50	0.0084	0.10
Hexachlorocyclopentadiene	8121(3510/3520)	2	D-111	≤50	0.0030	0.10
Hexachloroethane	8121(3510/3520)	2	8-139	≤50	0.0038	0.10
1,2,4-Trichlorobenzene	8121(3510/3520)	2	5-149	≤50	0.060	1.0
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8121(3510/3520)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	8121(3510/3520)	2	30-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RLA (ug/L)
Acephate(1)	1657	72	25-140	≤50	1.2	5.0
Methamidophos(1)	1657	72	36-106	≤46	0.10	2.0
Alachlor	8141(3510/3520)	2	46-139	≤30	0.14	1.0
Ametryn	8141(3510/3520)	2	60-120	≤40	0.16	2.0
Atrazine (MS)	8141(3510/3520)	2	39-130	≤30	0.13	2.0
Azinphos methyl	8141(3510/3520)	2	48-162	≤50	0.023	1.0
Benoxacor	8141(3510/3520)	2	10-150	≤50	4.4	10
Bolstar (Sulprofos)	8141(3510/3520)	2	36-114	≤40	0.012	1.0
Butachlor	8141(3510/3520)	2	50-150	≤40	0.35	1.0
Carbophenothion	8141(3510/3520)	2	69-122	≤40	0.23	1.0
Chlordimeform (Galecron)	8141(3510/3520)	2	10-150	≤50	3.8	10
5-Chloroaminotoluene	8141(3510/3520)	2	10-150	≤50	3.4	10
Chlorpyrifos	8141(3510/3520)	2	49-109	≤40	0.022	1.0
Chlorpyrifos methyl	8141(3510/3520)	2	53-136	≤40	0.17	1.0
Coumaphos	8141(3510/3520)	2	61-139	≤40	0.023	1.0
Demeton-o	8141(3510/3520)	2	10-117	≤40	0.030	2.5
Demeton-s	8141(3510/3520)	2	37-121	≤40	0.016	2.5
Diazinon (MS)	8141(3510/3520)	2	40-137	≤40	0.010	1.0
Dichlofenthion	8141(3510/3520)	2	38-118	≤40	0.11	1.0
Dichlorvos	8141(3510/3520)	2	11-158	≤40	0.047	2.0
Dimethoate	8141(3510/3520)	2	14-101	≤40	0.088	2.0
Dioxathion	8141(3510/3520)	2	26-127	≤40	1.5	10
Disulfoton	8141(3510/3520)	2	42-112	≤66	0.026	2.0
EPN	8141(3510/3520)	2	48-124	≤40	0.19	1.0
Ethion	8141(3510/3520)	2	62-175	≤40	0.12	0.50
Ethoprop	8141(3510/3520)	2	42-123	≤40	0.019	0.50
Famphur	8141(3510/3520)	2	13-128	≤60	0.25	2.0
Fenamiphos	8141(3510/3520)	2	40-160	≤40	0.59	2.0
Fensulfothion	8141(3510/3520)	2	31-153	≤40	0.035	5.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Fenthion	8141(3510/3520)	2	41-115	≤60	0.014	1.0
Isofenphos	8141(3510/3520)	2	40-160	≤40	0.060	0.50
Malathion	8141(3510/3520)	2	10-140	≤40	0.071	1.0
Merphos	8141(3510/3520)	2	32-138	≤40	0.22	1.0
Metolachlor	8141(3510/3520)	2	53-133	≤40	0.11	1.0
Metrabuzin	8141(3510/3520)	2	50-150	≤40	0.11	1.0
Mevinphos	8141(3510/3520)	2	24-166	≤40	0.022	2.0
Monocrotophos	8141(3510/3520)	2	43-126	≤50	2.9	10
Naled	8141(3510/3520)	2	10-119	≤40	0.022	5.0
Parathion, ethyl (MS)	8141(3510/3520)	2	28-155	≤34	0.083	1.0
Parathion, methyl (MS)	8141(3510/3520)	2	38-149	≤32	0.024	0.50
Phorate	8141(3510/3520)	2	28-119	≤40	0.027	1.0
Prometon	8141(3510/3520)	2	55-124	≤40	0.11	2.0
Prometryn	8141(3510/3520)	2	36-155	≤40	0.23	2.0
Propazine	8141(3510/3520)	2	51-127	≤30	0.25	2.0
Ronnel (MS)	8141(3510/3520)	2	30-130	≤35	0.021	1.0
Simazine	8141(3510/3520)	2	39-149	≤50	0.19	2.0
Stirophos (Tetrachlorvinphos)	8141(3510/3520)	2	48-125	≤40	0.014	1.0
Sulfotepp	8141(3510/3520)	2	40-157	≤40	0.17	0.50
Terbufos	8141(3510/3520)	2	40-160	≤40	0.16	1.0
Terbutylazine	8141(3510/3520)	2	60-130	≤40	0.33	2.0
Terbutryn	8141(3510/3520)	2	53-113	≤40	0.066	2.0
Thionazin (MS)	8141(3510/3520)	2	12-139	≤60	0.21	1.0
Tokuthion (Prothiofos)	8141(3510/3520)	2	45-114	≤40	0.22	1.0
Trichloronate	8141(3510/3520)	2	16-123	≤40	0.12	1.0
Surrogate - Triphenylphosphate	8141(3510/3520)	2	16-164	NA	NA	NA

(1) Determined by NPD

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL A (ug/L)
Bentazon	8151	2	50-150	≤48	0.24	2.0
2,4-D (MS)	8151	2	11-154	≤78	0.16	0.50
2,4-DB	8151	2	55-167	≤43	0.15	0.50
2,4,5-T (MS)	8151	2	25-128	≤48	0.087	0.50
2,4,5-TP (Silvex) (MS)	8151	2	10-100	≤66	0.048	0.50
Dalapon	8151	2	26-97	≤68	0.74	120
Dicamba	8151	2	38-152	≤46	0.070	1.2
Dichlorprop	8151	2	27-209	≤95	0.64	6.0
Dinoseb	8151	2	10-127	≤115	0.88	6.0
MCPA	8151	2	20-150	≤28	120	120
MCPP	8151	2	10-164	≤78	44	120
Pentachlorophenol	8151	2	11-110	≤34	0.043	1.0
Picloram	8151	2	10-150	≤56	0.092	0.50
Surrogate - 2,4-Dichlorophenylacetic acid (DCAA)	8151	2	27-133	NA	NA	NA
Surrogate - 2,4-Dichlorophenoxy butyric acid (2,4-DB)	8151	2	55-167	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Acetone	8260(5030)	2	32-174	≤52	9.9	50
	8260(5030)(low level)	2	32-174	≤52	12	25
Acetonitrile	8260(5030)	2	70-130	≤30	75	200
	8260(5030)(low level)	2	70-130	≤30	14	40
Acrolein	8260(5030)	2	54-145	≤74	43	100
	8260(5030) (low level)	2	54-145	≤74	9.2	20
Acrylonitrile	8260(5030)	2	10-183	≤41	9.7	100
	8260(5030) (low level)	2	10-183	≤1	5.0	20
Benzene (MS)	8260(5030)	2	62-135	≤37	0.27	5.0
	8260(5030)(low level)	2	62-135	≤37	0.10	1.0
Benzyl chloride	8260(5030)	2	70-130	≤30	25	100
	8260(5030)(low level)	2	70-130	≤30	12	50
Bromobenzene	8260(5030)	2	62-124	≤35	0.58	5.0
	8260(5030)(low level)	2	62-124	≤35	0.11	1.0
Bromochloromethane	8260(5030)	2	45-131	≤33	0.58	5.0
	8260(5030)(low level)	2	45-131	≤33	0.21	1.0
Bromodichloromethane	8260(5030)	2	65-125	≤28	0.35	5.0
	8260(5030)(low level)	2	65-125	≤28	0.14	1.0
Bromoform	8260(5030)	2	52-148	≤31	0.58	5.0
	8260(5030)(low level)	2	52-148	≤31	0.24	1.0
Bromomethane	8260(5030)	2	40-141	≤33	2.5	10
	8260(5030)(low level)	2	40-141	≤32	0.44	1.0
1,3-Butadiene	8260(5030)	2	70-130	≤30	1.0	10
2-Butanone (MEK)	8260(5030)	2	42-167	≤31	11	25
	8260(5030)(low level)	2	42-167	≤31	5.5	10
n-Butylbenzene	8260(5030)	2	51-117	≤33	0.67	5.0
	8260(5030)(low level)	2	51-117	≤33	0.14	1.0
sec-Butylbenzene	8260(5030)	2	41-150	≤34	0.63	5.0
	8260(5030)(low level)	2	41-150	≤34	0.11	1.0
tert-Butylbenzene	8260(5030)	2	42-141	≤34	0.84	5.0
	8260(5030)(low level)	2	42-141	≤34	0.070	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Carbon disulfide	8260(5030)	2	28-152	≤23	1.5	5.0
	8260(5030)(low level)	2	28-152	≤23	0.35	1.0
Carbon tetrachloride	8260(5030)	2	57-128	≤38	0.42	5.0
	8260(5030)(low level)	2	57-128	≤38	0.090	1.0
Chlorobenzene (MS)	8260(5030)	2	72-127	≤22	0.63	5.0
	8260(5030)(low level)	2	72-127	≤22	0.090	1.0
2-Chloro-1,3-butadiene	8260(5030)	2	70-130	≤30	0.89	5.0
(Chloroprene)	8260(5030)(low level)	2	70-130	≤30	0.17	1.0
Chloroethane	8260(5030)	2	47-148	≤34	1.6	10
	8260(5030)(low level)	2	47-148	≤34	0.22	1.0
2-Chloroethyl vinyl ether	8260(5030)	2	10-177	≤142	11	50
	8260(5030)(low level)	2	10-177	≤142	0.97	10
Chloroform	8260(5030)	2	62-130	≤20	0.90	5.0
	8260(5030)(low level)	2	62-130	≤20	0.14	1.0
Chloromethane	8260(5030)	2	34-145	≤44	2.1	10
	8260(5030)(low level)	2	34-145	≤44	0.30	1.0
3-Chloropropene (Allyl chloride)	8260(5030)	2	70-130	≤30	1.1	5.0
	8260(5030)(low level)	2	70-130	≤30	0.12	1.0
2-Chlorotoluene	8260(5030)	2	47-142	≤39	0.65	5.0
	8260(5030)(low level)	2	47-142	≤39	0.11	1.0
4-Chlorotoluene	8260(5030)	2	49-140	≤25	0.52	5.0
	8260(5030)(low level)	2	49-140	≤25	0.14	1.0
Cyclohexanone	8260(5030)(low level)	2	70-130	≤30	59	500
Dibromochloromethane	8260(5030)	2	68-126	≤31	0.51	5.0
	8260(5030)(low level)	2	68-126	≤31	0.17	1.0
1,2-Dibromo-3-chloropropane	8260(5030)	2	70-130	≤30	0.74	5.0
(DBCP)	8260(5030)(low level)	2	70-130	≤30	0.38	1.0
1,2-Dibromoethane	8260(5030)	2	45-137	≤40	0.50	5.0
	8260(5030)(low level)	2	45-137	≤40	0.13	1.0
Dibromomethane	8260(5030)	2	62-130	≤40	0.41	5.0
	8260(5030)(low level)	2	62-130	≤40	0.070	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
1,2-Dichlorobenzene	8260(5030)	2	57-129	≤39	0.44	5.0
	8260(5030)(low level)	2	57-129	≤39	0.080	1.0
1,3-Dichlorobenzene	8260(5030)	2	52-140	≤29	0.64	5.0
	8260(5030)(low level)	2	52-140	≤29	0.11	1.0
1,4-Dichlorobenzene	8260(5030)	2	51-138	≤31	0.52	5.0
	8260(5030)(low level)	2	51-138	≤31	0.14	1.0
trans-1,4-Dichloro-2-butene	8240(5030)/8260(5030)	2	70-130	≤30	2.5	10
	8260(5030)(low level)	2	70-130	≤30	1.2	2.0
Dichlorodifluoromethane	8260(5030)	2	30-126	≤34	1.2	10
	8260(5030)(low level)	2	30-126	≤34	0.20	1.0
1,1-Dichloroethane	8260(5030)	2	51-140	≤47	0.52	5.0
	8260(5030)(low level)	2	51-140	≤47	0.38	1.0
1,2-Dichloroethane	8260(5030)	2	65-131	≤23	0.57	5.0
	8260(5030)(low level)	2	65-131	≤23	0.46	1.0
Dichloroethenes (Total)	8260(5030)	2	43-136	≤22	0.44	5-0
(sum of cis- and trans- isomers)	8260(5030)(low level)	2	43-136	≤22	0.19	1.0
cis-1,2-Dichloroethene	8240(5030)/8260	2	50-128	≤28	0.65	5.0
	8240(5030)/8260(5030)(low level)	2	50-128	≤28	0.51	1.0
trans-1,2-Dichloroethene	8260	2	43-136	≤22	0.44	5.0
	8260(5030)(low level)	2	43-136	≤22	0.19	1.0
1,1-Dichloroethene	8260(5030)	2	46-147	≤30	0.45	5.0
	8260(5030)(low level)	2	46-147	≤30	0.19	1.0
1,2-Dichloropropane	8260(5030)	2	67-128	≤24	0.52	5.0
	8260(5030)(low level)	2	67-128	≤24	0.090	1.0
1,3-Dichloropropane	8260(5030)	2	49-139	≤40	0.39	5.0
	8260(5030)(low level)	2	49-139	≤40	0.14	1.0
2,2-Dichloropropane	8260(5030)	2	46-119	≤21	1.1	5.0
	8260(5030)(low level)	2	46-119	≤21	0.40	1.0
1,1-Dichloropropene	8260(5030)	2	54-114	0-37	0.31	5.0
	8260(5030)(low level)	2	54-114	0-37	0.12	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
cis-1,3-Dichloropropene	8260(5030)	2	66-125	≤21	0.47	5.0
	8260(5030)(low level)	2	66-125	≤21	0.17	1.0
trans-1,3-Dichloropropene	8260(5030)	2	49-136	≤24	0.38	5.0
	8260(5030)(low level)	2	49-136	≤24	0.49	1.0
Diethyl ether	8260(5030)	2	70-130	≤30	2.5	10
	8260(5030)(low level)	2	70-130	≤30	0.50	2.0
Ethanol	8260(5030)(low level)	2	70-130	≤30	250	1000
Ethyl acetate	8260(5030)	2	70-130	≤30	2.5	10
Ethylbenzene	8260(5030)	2	74-122	≤18	0.83	5.0
	8260(5030)(low level)	2	74-122	≤18	0.11	1.0
Ethyl methacrylate	8260(5030)	2	70-130	≤30	0.53	5.0
	8260(5030)(low level)	2	70-130	≤30	0.42	1.0
Ethylene oxide	8240(5030)/8260(5030)(low level)	2	70-130	≤30	99	120
Hexachlorobutadiene	8260(5030)	2	55-127	≤29	2.3	5.0
	8260(5030)(low level)	2	55-127	≤29	0.14	1.0
Hexane	8260(5030)	2	70-130	≤30	1.2	5.0
2-Hexanone	8260(5030)	2	48-155	≤36	4.4	25
	8260(5030)(low level)	2	48-155	≤36	1.5	10
Iodomethane	8260(5030)	2	70-130	≤30	0.67	5.0
	8260(5030)(low level)	2	70-130	≤30	0.37	1.0
Isobutyl alcohol	8260(5030)	2	70-130	≤30	31	200
	8260(5030)(low level)	2	70-130	≤30	11	40
Isopropylbenzene	8260(5030)	2	45-136	≤36	0.95	5.0
	8260(5030)(low level)	2	45-136	≤36	0.10	1.0
p-Isopropyltoluene	8260(5030)	2	52-132	≤34	0.69	5.0
	8260(5030)(low level)	2	52-132	≤34	0.13	1.0
Methacrylonitrile	8260(5030)	2	70-130	≤30	8.4	100
	8260(5030)(low level)	2	70-130	≤30	8.1	20
Methylene chloride	8260(5030)	2	47-140	≤50	0.31	5.0
(Dichloromethane)	8260(5030)(low level)	2	47-140	≤50	0.25	5.0
Methylmethacrylate	8240(5030)	2	70-130	≤30	0.66	5.0
	8240(5030)(low level)	2	70-130	≤30	0.55	1.0
4-Methyl-2-pentanone (MIBK)	8260(5030)	2	50-150	≤42	8.6	25
	8260(5030)(low level)	2	50-150	≤42	0.59	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Methyl t-butyl ether (MTBE)	8260(5030)	2	56-124	≤38	1.5	10
	8260(5030)(low level)	2	56-124	≤38	0.85	10
Naphthalene	8260(5030)	2	40-139	≤50	2.3	5.0
	8260(5030)(low level)	2	40-139	≤50	0.21	1.0
n-Octane	8260	2	70-130	≤30	1.2	5.0
Pentachloroethane	8260(5030)	2	70-130	≤30	11	25
	8260(5030)(low level)	2	70-130	≤30	4.2	5.0
Propionitrile (ethylcyanide)	8260(5030)	2	70-130	≤30	7.2	100
	8260(5030)(low level)	2	70-130	≤30	13	20
n-Propylbenzene	8260(5030)	2	56-122	≤34	0.59	5.0
	8260(5030)(low level)	2	56-122	≤34	0.19	1.0
Styrene	8260(5030)	2	66-130	≤28	0.98	5.0
	8260(5030)(low level)	2	66-130	≤28	0.16	1.0
1,1,1,2-Tetrachloroethane	8260(5030)	2	59-137	≤20	0.63	5.0
	8260(5030)(low level)	2	59-137	≤20	0.11	1.0
1,1,2,2-Tetrachloroethane	8260(5030)	2	67-133	≤22	0.75	5.0
	8260(5030)(low level)	2	67-133	≤22	0.13	1.0
Tetrachloroethene	8260(5030)	2	60-148	≤24	1.6	5.0
	8260(5030)(low level)	2	60-148	≤24	0.38	1.0
Toluene (MS)	8260(5030)	2	68-131	≤33	0.51	5.0
	8260(5030)(low level)	2	68-131	≤33	0.28	1.0
1,2,3-Trichlorobenzene	8260(5030)	2	30-140	≤65	0.77	5.0
	8260(5030)(low level)	2	30-140	≤65	0.17	1.0
1,2,4-Trichlorobenzene	8260(5030)	2	40-147	≤57	0.58	5.0
	8260(5030)(low level)	2	40-147	≤57	0.16	1.0
1,1,1-Trichloroethane	8260(5030)	2	69-120	≤27	0.46	5.0
	8260(5030)(low level)	2	69-120	≤27	0.14	1.0
1,1,2-Trichloroethane	8260(5030)	2	63-133	≤21	0.47	5.0
	8260(5030)(low level)	2	63-133	≤21	0.15	1.0
Trichloroethene(MS)	8260(5030)	2	56-143	≤35	0.28	5.0
	8260(5030)(low level)	2	56-143	≤35	0.17	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Trichlorofluoromethane	8260(5030)	2	53-136	≤21	0.98	5.0
	8260(5030)(low level)	2	53-136	≤21	0.49	1.0
1,2,3-Trichloropropane	8260(5030)	2	67-122	≤26	1.3	5.0
	8260(5030)(low level)	2	67-122	≤26	0.49	1.0
1,1,2-Trichloro-1,2,2-	8260(5030)	2	70-130	≤30	0.26	5.0
Trifluoroethane	8260(5030)(low level)	2	70-130	≤30	0.57	1.0
1,2,4-Trimethylbenzene	8260(5030)	2	61-133	≤20	0.86	5.0
	8260(5030)(low level)	2	61-133	≤20	0.090	1.0
1,3,5-Trimethylbenzene	8260(5030)	2	47-138	≤48	1.1	5.0
	8260(5030)(low level)	2	47-138	≤48	0.15	1.0
Vinyl acetate	8260(5030)	2	10-166	≤22	1.5	10
	8260(5030)(low level)	2	10-166	≤22	0.15	2.0
Vinyl chloride	8260(5030)	2	43-142	≤21	0.50	10
	8260(5030)(low level)	2	43-142	≤21	0.28	2.0
Xylenes (total)	8260(5030)	2	73-135	≤26	1.9	10
	8260(5030) (low level)	2	73-135	≤26	0.31	2.0
o-Xylene	8260(5030)	2	72-129	≤26	0.78	5.0
	8260(5030)(low level)	2	72-129	≤26	0.12	1.0
m+p-Xylene	8260(5030)	2	73-135	≤26	1.9	5.0
	8260(5030)(low level)	2	73-135	≤26	0.31	1.0
Toluene-d8	8260(5030)	2	77-122	NA	NA	NA
(Surrogate)	8260(5030)(low level)	2	77-122	NA	NA	NA
p-Bromofluorobenzene	8260(5030)	2	74-126	NA	NA	NA
(Surrogate)	8260(5030)(low level)	2	74-126	NA	NA	NA
Dibromofluoromethane	8260(5030)	2	70-130	NA	NA	NA
(Surrogate)	8260(5030)(low level)	2	70-130	NA	NA	NA
1,2-Dichloroethane-d4	8240(5030)	2	70-130	NA	NA	NA
(Surrogate)	8240(5030)(low level)	2	70-130	NA	NA	NA
1,2-Dichlorobenzene-d4	8260(5030)	2	70-130	NA	NA	NA
(Surrogate)	8260(5030)(low level)	2	70-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Acenaphthene (MS)	8270(3510/3520)	2	36-121	≤35	0.25	10
	8270(3510/3520)-low level	2	36-121	≤35	0.088	0.20
Acenaphthylene	8270(3510/3520)	2	41-121	≤28	0.33	10
	8270(3510/3520)-low level	2	41-121	≤28	0.074	0.20
Acetophenone	8270(3510/3520)	2	10-150	≤50	0.52	10
2-Acetylaminofluorene	8270(3510/3520)	2	25-150	≤50	0.50	10
Aldrin	8270(3510/3520)	2	62-119	≤21	0.34	10
4-Aminobiphenyl	8270(3510/3520)	2	10-150	≤50	4.8	10
Aniline	8270(3510/3520)	2	10-180	≤32	4.2	20
Anthracene	8270(3510/3520)	2	45-126	≤21	0.33	10
	8270(3510/3520)-low level	2	45-126	≤21	0.076	0.20
Aramite	8270(3510/3520)	2	40-150	≤50	1.1	10
Benzidine	8270(3510/3520)	2	10-133	≤121	5.6	80
Benzoic acid	8270(3510/3520)	2	10-150	≤66	9.9	50
Benzo(a)anthracene	8270(3510/3520)	2	33-136	≤34	0.30	10
	8270(3510/3520)-low level	2	33-136	≤34	0.060	0.20
	SIM	73	64-100	≤25	0.042	0.10
Benzo(b)fluoranthene	8270(3510/3520)	2	33-132	≤32	0.28	10
	8270(3510/3520)-low level	2	33-132	≤32	0.057	0.20
	SIM	73	60-103	≤25	0.036	0.10
Benzo(k)fluoranthene	8270(3510/3520)	2	33-150	≤34	0.72	10
	8270(3510/3520)-low level	2	33-150	≤34	0.057	0.20
	SIM	73	63-101	≤25	0.038	0.10
p-Benzoquinone	8270(3510/3520)	2	40-140	≤40	3.7	10
Benzo(g,h,i)perylene	8270(3510/3520)	2	28-146	≤39	0.68	10
	8270(3510/3520)-low level	2	28-146	≤39	0.056	0.20
Benzo(a)pyrene	8270(3510/3520)	2	45-120	≤24	0.41	10
	8270(3510/3520)-low level	2	45-120	≤24	0.057	0.20
	SIM	73	48-100	≤25	0.036	0.10
Benzyl alcohol	8270(3510/3520)	2	13-134	≤32	0.49	10
Benzyl chloride	8270(3510/3520)	2	10-150	≤50	2.5	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Alpha-BHC	8270(3510/3520)	2	44-131	≤30	0.37	10
Beta-BHC	8270(3510/3520)	2	29-127	≤33	0.80	10
Delta-BHC	8270(3510/3520)	2	10-161	≤44	1.7	10
Gamma-BHC	8270(3510/3520)	2	43-120	≤27	0.30	10
Biphenyl (Diphenyl)	8270(3510/3520)	2	50-130	≤25	2.2	10
Bis(2-chloroethoxy) methane	8270(3510/3520)	2	47-110	≤20	0.26	10
Bis(2-chloroethyl) ether	8270(3510/3520)	2	34-114	≤58	0.44	10
Bis(2-chloroisopropyl) ether (2,2-Oxybis(1-chloropropane)	8270(3510/3520)	2	15-144	≤23	0.23	10
Bis(2-ethylhexyl) phthalate	8270(3510/3520)	2	34-160	≤26	0.48	10
4-Bromophenyl phenyl ether	8270(3510/3520)	2	36-124	≤26	0.35	10
Butyl benzyl phthalate	8270(3510/3520)	2	38-141	≤41	0.41	10
Carbazole	8270(3510/3520)	2	48-127	≤23	0.54	10
4-Chloroaniline	8270(3510/3520)	2	10-130	≤67	0.98	20
4-Chloro-3-methyl-phenol (p-Chloro-m-cresol)(MS)	8270(3510/3520)	2	34-126	≤31	0.33	10
1-Chloronaphthalene	8270(3510/3520)	2	45-107	≤22	2.5	10
2-Chloronaphthalene	8270(3510/3520)	2	45-107	≤22	0.39	10
2-Chlorophenol (MS)	8270(3510/3520)	2	38-115	≤34	0.24	10
4-Chlorophenylphenyl ether	8270(3510/3520)	2	22-140	≤26	0.66	10
Chrysene	8270(3510/3520)	2	44-128	≤31	0.44	10
	8270(3510/3520)-low level	2	44-128	≤31	0.069	0.20
	SIM	73	72-109	≤25	0.044	0.10
1,8-Cineole	8270(3510/3520)	2	50-101	≤25	0.80	10
p-Cymene	8270(3510/3520)	2	52-110	≤25	1.6	10
o-Cresol (2-Methyl phenol)	8270(3510/3520)	2	31-119	≤33	0.29	10
m- Cresol (3 -Methyl phenol)	8270(3510/3520)	2	24-136	≤27	0.71	10
p-Cresol (4-Methyl phenol)	8270(3510/3520)	2	24-136	≤27	0.71	10
m- and p-Cresols(3 and 4-Methyl phenol)	8270(3510/3520)	2	24-136	≤27	0.71	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
4,4'-DDD	8270(3510/3520)	2	16-124	≤50	0.35	10
4,4'-DDE	8270(3510/3520)	2	30-125	≤35	0.30	10
4,4'-DDT	8270(3510/3520)	2	22-119	≤26	0.40	10
Diallate	8270(3510/3520)	2	10-150	≤50	1.2	10
Dibenz(a,h)anthracene	8270(3510/3520)	2	33-143	≤35	0.80	10
	8270(3510/3520)-low level	2	33-143	≤35	0.054	0.20
	SIM	73	47-117	≤25	0.042	0.10
Dibenzofuran	8270(3510/3520)	2	50-119	≤20	0.29	10
Dibenzo(a,e)pyrene	8270(3510/3520)	2	29-177	≤60	2.5	10
Di-n-butyl phthalate	8270(3510/3520)	2	43-145	≤29	0.26	10
1,2-Dichlorobenzene	8270(3510/3520)	2	34-130	≤30	0.31	10
1,3-Dichlorobenzene	8270(3510/3520)	2	28-130	≤26	0.32	10
1,4-Dichlorobenzene(MS)	8270(3510/3520)	2	27-130	≤31	0.29	10
3,3'-Dichlorobenzidine	8270(3510/3520)	2	10-144	≤72	4.4	20
2,4-Dichlorophenol	8270(3510/3520)	2	25-134	≤30	0.66	10
2,6-Dichlorophenol	8270(3510/3520)	2	10-150	≤50	1.0	10
Dieldrin	8270(3510/3520)	2	29-133	≤20	0.41	10
Di(2-ethylhexyl)adipate	8270(3520)	2	35-130	≤50	0.36	10
Diethyl phthalate	8270(3510/3520)	2	46-134	≤49	0.47	10
Dimethoate	8270(3510/3520)	2	10-150	≤50	2.5	10
p-(Dimethylamino)azobenzene	8270(3510/3520)	2	10-150	≤50	1.3	10
7,12-Dimethylbenz(a)anthracene	8270(3510/3520)	2	10-150	≤50	2.7	10
3,3'-Dimethylbenzidine	8270(3510/3520)	2	10-200	≤100	13	20
a,a-Dimethylphenethylamine	8270(3510/3520)	2	10-200	≤50	9.7	2000
2,4-Dimethylphenol	8270(3510/3520)	2	28-130	≤43	0.39	10
Dimethylphthalate	8270(3510/3520)	2	22-141	≤31	0.39	10
m-Dinitrobenzene	8270(3510/3520)	2	10-150	≤50	0.91	10
p-Dinitrobenzene	8270(3510/3520)	2	44-125	≤58	8.0	50
4,6-Dinitro-2-methylphenol	8270(3510/3520)	2	10-173	≤33	5.0	50

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL ^ (ug/L)
2,4-Dinitrophenol	8270(3510/3520)	2	10-209	≤63	0.80	50
2,4-Dinitrotoluene (MS)	8270(3510/3520)	2	37-129	≤32	0.41	10
2,6-Dinitrotoluene	8270(3510/3520)	2	24-139	≤24	0.34	10
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270(3510/3520)	2	10-150	≤50	1.1	10
Di-n-octyl phthalate	8270(3510/3520)	2	24-152	≤33	0.35	10
Diphenylamine	8270(3510/3520)	2	15-135	≤50	5.4	10
1,4-Dioxane	8270(3510/3520)	2	10-150	≤50	1.0	10
1,2-Diphenyl hydrazine	8270(3510/3520)	2	25-130	≤32	0.81	10
Disulfoton	8270(3510/3520)	2	10-150	≤50	2.5	10
Endosulfan I	8270(3510/3520)	2	15-135	≤68	1.8	20
Endosulfan II	8270(3510/3520)	2	22-118	≤60	1.3	20
Endosulfan sulfate	8270(3510/3520)	2	37-118	≤34	0.75	20
Endrin	8270(3510/3520)	2	21-125	≤50	2.3	20
Endrin aldehyde	8270(3510/3520)	2	10-234	≤59	18	50
Endrin ketone	8270(3510/3520)	2	10-150	≤50	12	50
Ethyl carbamate	8270(3510/3520)	2	52-100	≤25	2.5	10
Ethyl methanesulfonate	8270(3510/3520)	2	10-150	≤50	0.70	10
Ethyl parathion	8270(3510/3520)	2	10-150	≤50	2.5	10
Famphur	8270(3510/3520)	2	10-150	≤50	2.5	10
Fluoranthene	8270(3510/3520)	2	41-129	≤24	0.33	10
	8270(3510/3520)-low level	2	41-129	≤24	0.054	0.20
Fluorene	8270(3510/3520)	2	50-124	≤23	0.38	10
	8270(3510/3520)-low level	2	50-124	≤23	0.087	0.20
Heptachlor	8270(3510/3520)	2	17-130	≤27	0.42	20
Heptachlor epoxide	8270(3510/3520)	2	34-130	≤29	0.58	20
Hexachlorobenzene	8270(3510/3520)	2	49-121	≤31	0.19	10
Hexachlorobutadiene	8270(3510/3520)	2	27-130	≤30	0.35	10
Hexachlorocyclopentadiene	8270(3510/3520)	2	D-130	≤67	2.4	10
Hexachloroethane	8270(3510/3520)	2	26-130	≤35	0.32	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Hexachlorophene(1)	8270(3510/3520)	2	10-200	≤80	29	5000
Hexachloropropene	8270(3510/3520)	2	10-150	≤50	0.53	10
Indeno(1,2,3-cd)pyrene	8270(3510/3520)	2	37-140	≤38	0.56	10
	8270(3510/3520)-low level	2	37-140	≤38	0.064	0.20
	SIM	73	38-100	≤35	0.043	0.10
Isophorone	8270(3510/3520)	2	29-130	≤33	0.37	10
Isosafrole	8270(3510/3520)	2	10-150	≤50	0.64	10
Kepone	8270(3510/3520)	2	10-150	≤50	5.3	10
d-Limonene	8270(3510/3520)	2	36-74	≤25	1.4	10
Methapyrilene	8270(3510/3520)	2	10-150	≤50	1.6	2000
4,4-Methylbis(2-chloroaniline)	8270(3510/3520)	2	39-121	≤73	92	500
3-Methylcholanthrene	8270(3510/3520)	2	10-150	≤50	0.76	10
Methylmethanesulfonate	8270(3510/3520)	2	10-150	≤50	0.72	10
2-Methylnaphthalene	8270(3510/3520)	2	43-130	≤30	0.33	10
	8270(3510/3520)-low level	2	43-130	≤30	0.054	0.20
1-Methylnaphthalene	8270(3510/3520)	2	35-130	≤30	0.68	10
	8270(3510/3520)-low level	2	35-130	≤30	0.073	0.20
Methyl parathion	8270(3510/3520)	2	10-150	≤50	2.5	10
2,4(8)-p-Menthadiene	8270(3510/3520)	2	22-85	≤42	1.6	10
Naphthalene	8270(3510/3520)	2	41-130	≤33	0.36	10
	8270(3510/3520)-low level	2	41-130	≤33	0.071	0.20
1,4-Naphthoquinone	8270(3510/3520)	2	10-150	≤50	0.78	10
1-Naphthylamine	8270(3510/3520)	2	10-150	≤50	1.1	10
2-Naphthylamine	8270(3510/3520)	2	10-150	≤50	0.98	10
Nicotine	8270(3510/3520)	2	10-150	≤50	25	100
2-Nitroaniline	8270(3510/3520)	2	26-130	≤49	5.3	50
3-Nitroaniline	8270(3510/3520)	2	10-130	≤57	5.0	50
4-Nitroaniline	8270(3510/3520)	2	10-140	≤39	7.7	50
Nitrobenzene	8270(3510/3520)	2	50-111	≤21	0.31	10
2-Nitrophenol	8270(3510/3520)	2	35-125	≤24	0.36	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
4-Nitrophenol (MS)	8270(3510/3520)	2	12-143	≤44	4.9	50
4-Nitroquinoline-1-oxide	8270(3510/3520)	2	10-150	≤50	2.5	20
N-Nitrosodi-n-butylamine	8270(3510/3520)	2	10-150	≤50	0.62	10
N-Nitrosodiethylamine	8270(3510/3520)	2	10-150	≤50	0.46	10
N-Nitrosodimethylamine	8270(3510/3520)	2	10-133	≤31	0.49	10
N-Nitrosodi-n-propylamine(MS)	8270(3510/3520)	2	31-138	≤30	0.29	10
N-nitrosodiphenylamine/ Diphenylamine	8270(3510/3520)	2	24-146	≤25	0.34	10
N-Nitrosodiphenylamine	8270(3510/3520)	2	24-146	≤25	0.34	10
N-Nitrosomethylethylamine	8270(3510/3520)	2	10-150	≤50	8.6	10
N-Nitrosomorpholine	8270(3510/3520)	2	10-150	≤50	1.1	10
N-Nitrosopiperidine	8270(3510/3520)	2	10-150	≤50	1.6	10
N-Nitrosopyrrolidine	8270(3510/3520)	2	10-150	≤50	0.86	10
5-Nitro-o-toluidine	8270(3510/3520)	2	10-150	≤50	1.3	10
Pentachlorobenzene	8270(3510/3520)	2	10-150	≤50	0.68	10
Pentachloronitrobenzene	8270(3520)0	2	10-150	≤50	1.3	10
Pentachlorophenol (MS)	8270(3510/3520)	2	19-148	≤33	4.0	50
	SIM	73	10-102	≤50	0.60	1.0
beta-Phellandrene	8270(3510/3520)	2	33-65	≤25	1.9	10
Phenacetin	8270(3510/3520)	2	10-150	≤50	1.2	10
Phenanthrene	8270(3510/3520)	2	50-121	≤20	0.33	10
	8270(3510/3520)-low level	2	50-121	≤20	0.089	0.20
Phenol (MS)	8270(3510/3520)	2	33-122	≤36	0.28	10
Phenyl ether (Diphenyl oxide)	8270(3510/3520)	2	74-116	≤25	2.0	10
p-Phenylenediamine	8270(3510/3520)	2	10-200	≤50	500	2000
Phorate	8270(3510/3520)	2	10-150	≤50	2.5	10
2-Picoline	8270(3510/3520)	2	10-150	≤50	1.1	10
cis-Pinane	8270(3510/3520)	2	50-103	≤29	2.9	10
alpha-Pinene	8270(3510/3520)	2	12-105	≤25	1.3	100
beta-Pinene	8270(3510/3520)	2	DL-155	≤56	2.0	100

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Pronamide	8270(3510/3520)	2	10-150	≤50	0.56	10
Pyrene(MS)	8270(3510/3520)	2	31-139	≤42	0.53	10
	8270(3510/3520)-low level	2	31-139	≤42	0.089	0.20
Pyridine	8270(3510/3520)	2	D-134	≤50	0.93	50
Resorcinol	8270 (3510/3520)	2	6-110	≤54	2.2	200
Safrole	8270(3510/3520)	2	10-150	≤50	0.95	10
Strychnine	8270(3510/3520)	2	10-150	≤50	25	100
Sulfotepp	8270(3510/3520)	2	10-150	≤50	2.5	10
alpha-Terpinene	8270(3510/3520)	2	DL-94	≤96	1.2	10
Terpineol	8270(3510/3520)	2	50-150	≤50	0.82	10
Terpinolene	8270(3510/3520)	2	20-150	≤45	1.7	10
1,2,4,5-Tetrachlorobenzene	8270(3510/3520)	2	10-150	≤50	0.75	10
2,3,4,5-Tetrachlorophenol	8270(3510/3520)	2	10-150	≤50	0.61	10
2,3,4,6-Tetrachlorophenol	8270(3510/3520)	2	34-130	≤31	0.57	10
Tetrachlorophenols (2,3,4,5 and 2,3,4,6)	8270(3510/3520)	2	10-150	≤50	0.61	10
Thionazin	8270(3510/3520)	2	10-150	≤50	2.5	10
o-Toluidine	8270(3510/3520)	2	10-150	≤50	0.53	10
1,2,4-Trichlorobenzene (MS)	8270(3510/3520)	2	28-110	≤28	0.36	10
2,4,5-Trichlorophenol	8270(3510/3520)	2	38-127	≤28	0.74	10
2,4,6-Trichlorophenol	8270(3510/3520)	2	36-126	≤22	0.35	10
Trichlorophenols (2,4,5 and 2,4,6)	8270(3510/3520)	2	38-127	≤28	0.74	10
o,o,o-Triethyl-phosphorothioate	8270(3510/3520)	2	10-150	≤50	1.6	10
1,3,5-Trinitrobenzene	8270(3510/3520)	2	10-150	≤50	1.5	10
Nitrobenzene-d5 (Surrogate)	8270(3510/3520)	2	34-130	NA	NA	NA
2-Fluorobiphenyl (Surrogate)	8270(3510/3520)	2	36-124	NA	NA	NA
Terphenyl-d14 (Surrogate)	8270(3510/3520)	2	14-148	NA	NA	NA
Phenol-d5 (Surrogate)	8270(3510/3520)	2	25-128	NA	NA	NA
2-Fluorophenol (Surrogate)	8270(3510/3520)	2	29-121	NA	NA	NA
2,4,6-Tribromophenol (Surrogate)	8270(3510/3520)	2	29-143	NA	NA	NA
Orhto Terphenyl (Surrogate)	8270(3520)-low level	2	30-130	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/L)	RLA (ug/L)
Polychlorinated Dibenzo-p-dioxin 2,3,7,8- substituted Congeners						
2,3,7,8-TCDD	8280	2	47-155	≤40	0.00070	0.0050
Polychlorinated Dibenzo-p-dioxin and Dibenzofuran classes						
tetra-CDD (MS)	8280	2	47-155	≤40	0.00070	0.0050
tetra-CDF (MS)	8280	2	67-154	≤40	0.00060	0.0050
penta-CDD (MS)	8280	2	50-168	≤40	0.0012	0.0050
Penta-CDF (MS)	8280	2	71-158	≤40	0.0011	0.0050
hexa-CDD (MS)	8280	2	72-164	≤40	0.0012	0.0050
hexa-CDF (MS)	8280	2	72-175	≤40	0.0013	0.0050
hepta-CDD (MS)	8280	2	20-170	≤50	0.0016	0.010
hepta-CDF (MS)	8280	2	20-170	≤50	0.00060	0.010
octa-CDD (MS)	8280	2	20-170	≤50	0.0057	0.010
octa-CDF (MS)	8280	2	20-170	≤50	0.0020	0.010
Internal Standards						
2,3,7,8-tetra-CDD-13C12	8280	2	25-150	NA	NA	NA
octa-CDD-13C12	8280	2	25-150	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RLA (ug/L)
Acenaphthene (MS)	8310(3510/3520)	2	32-128	≤37	0.11	1.0
Acenaphthylene	8310(3510/3520)	2	16-130	≤56	0.11	1.0
Anthracene	8310(3510/3520)	2	28-126	≤47	0.0049	0.20
Benzo(a)anthracene	8310(3510/3520)	2	40-122	≤25	0.0061	0.20
Benzo(b)fluoranthene	8310(35103520)	2	21-140	≤38	0.0037	0.20
Benzo(k)fluoranthene	8310(3510/3520)	2	23-135	≤42	0.011	0.20
Benzo(g,h,I)perylene	8310(3510/3520)	2	18-126	≤36	0.013	0.50
Benzo(a)pyrene	8310(3510/3520)	2	23-138	≤31	0.0075	0.20
Carbazole	8310 (3510/3520)	2	10-150	≤40	0.25	1.0
Chrysene (MS)	8310(3510/3520)	2	40-122	≤26	0.0066	0.20
Dibenzo(a,h)anthracene	8310(3510/3520)	2	14-126	≤45	0.036	0.20
Fluoranthene	8310(3510/3520)	2	37-136	≤68	0.014	0.50
Fluorene (MS)	8310(3510/3520)	2	31-130	≤45	0.020	0.50
Indeno(1,2,3-cd)pyrene	8310(3510/3520)	2	20-131	≤49	0.011	0.20
1-Methylnaphthalene	8310(3510/3520)	2	31-112	≤30	0.086	1.0
2-Methylnaphthalene	8310(3510/3520)	2	33-130	≤50	0.068	1.0
Naphthalene (MS)	8310(3510/3520)	2	15-130	≤35	0.056	1.0
Phenanthrene	8310(3510/3520)	2	37-122	≤50	0.013	0.20
Pyrene (MS)	8310(3510/3520)	2	29-137	≤40	0.012	0.50
Surrogate - 4-Terphenyl-d14	8310(3510/3520)	2	32-141	NA	NA	NA

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Acetaldehyde	8315	2	30-110	≤40	2.9	200
Formaldehyde	8315	2	43-142	≤30	3.5	50
Acrylamide	8316	2	59-136	≤30	5.9	100
Acrylonitrile	8316	2	60-140	≤30	26	200
Acrolein	8316	2	60-140	≤30	49	400
Aldicarb (Temik) (MS)	8318	2	34-124	≤40	0.0090	1.0
Aldicarb sulfone	8318	2	54-116	≤40	0.0089	1.0
Aldicarb sulfoxide	8318	2	17-51	≤49	0.0047	1.0
Carbaryl (Sevin)	8318	2	55-125	≤40	0.012	1.0
Carbofuran (Furadan) (MS)	8318	2	17-77	≤40	0.020	1.0
Dioxacarb	8318	2	56-124	≤40	0.013	1.0
3-Hydroxycarbofuran	8318	2	47-123	≤40	0.032	1.0
Methiocarb (Mesurol)	8318	2	51-137	≤40	0.0064	1.0
Methomyl (Lannate)	8318	2	57-125	≤40	0.017	1.0
Oxamyl (MS)	8318	2	31-103	≤40	0.011	1.0
Promecarb	8318	2	48-122	≤40	0.014	1.0
Propoxur (Baygon)	8318	2	47-127	≤40	0.020	1.0

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
2-Amino-4,6-dinitrotoluene-Low Level	8330	2/109	60-128	≤39	0.11	0.30
2-Amino-4,6-dinitrotoluene-High Level	8330	2	60-128	≤39	16	100
4-Amino-2,6-dinitrotoluene-Low Level	8330	2/109	35-155	≤57	0.13	0.50
4-Amino-2,6-dinitrotoluene-High Level	8330	2	35-155	≤57	20	100
1,3-Dinitrobenzene (MS)-Low Level	8330	2/109	71-130	≤17	0.040	0.30
1,3-Dinitrobenzene (MS)-High Level	8330	2	71-130	≤17	5.2	50
2,4-Dinitrotoluene (MS)-Low Level	8330	2/109	63-130	≤28	0.053	0.30
2,4-Dinitrotoluene (MS)-High Level	8330	2	63-130	≤28	14	100
2,6-Dinitrotoluene-Low Level	8330	2/109	61-130	≤34	0.088	0.50
2,6-Dinitrotoluene-High Level	8330	2	61-130	≤34	25	100
Diphenylamine-High Level	8330	2	65-140	≤30	1.0	10
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)-Low Level	8330	2/109	41-154	≤47	0.14	1.0
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)-High Level	8330	2	41-154	≤47	15	100
Methyl-2,4,6-trinitro-phenylnitramine (Tetryl)-Low Level	8330	2/109	37-184	≤67	0.14	0.50
Methyl-2,4,6-trinitro-phenylnitramine (Tetryl)-High Level	8330	2	37-184	≤67	13	100
Nitrobenzene-Low Level	8330	2/109	33-125	≤44	0.13	0.30
Nitrobenzene-High Level	8330	2	33-125-	≤44	10	50
Nitroglycerin-High Level	8332/8332	2	43-137	≤22	5.0	30
n-Nitrosodiphenylamine-High Level	8330	2	55-121	≤30	2.1	10
2-Nitrotoluene (MS)-Low Level	8330	2/109	52-133	≤39	0.087	0.50
2-Nitrotoluene (MS)-High Level	8330	2	52-133	≤39	22	100
3-Nitrotoluene-Low Level	8330	2/109	25-154	≤30	0.23	0.50
3-Nitrotoluene-High Level	8330	2	25-154	≤30	35	100
4-Nitrotoluene-Low Level	8330	2/109	39-151	≤40	0.27	0.50
4-Nitrotoluene-High Level	8330	2	39-151	≤40	50	100
Octahydro-1,3,5,7-tetranitro-1,3,5,7-Tetrazocine (HMX)-Low Level	8330	2/109	43-164	≤26	θ37	1.0
Octahydro-1,3,5,7-tetranitro-1,3,5,7-Tetrazocine (HMX)-High Level	8330	2	43-164	≤26	15	100
Pentaerythritol tetranitrate (PETN)-High Level	8330	2	41-159	≤30	5.9	20

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
1,3,5-Trinitrobenzene-Low Level	8330	2/109	24-155	≤17	0.059	0.30
1,3,5-Trinitrobenzene-High Level	8330	2	24-155	≤17	9.8	50
2,4,6-Trinitrotoluene-Low Level	8330	2/109	73-129	≤34	0.068	0.30
2,4,6-Trinitrotoluene-High Level	8330	2	73-129	≤34	15	100
Surrogate - 3,4-Dinitrotoluene	8330	2/109	26-165	NA.	NA	NA

The Low Level analysis of Explosives by 8330 and 8332 may be performed using SW-846 Extraction Method 3520 in place of the routine "salting out" extraction described in SW-846 Method 8330. This modification is described in SL SOP LC41-T(reference 109).

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL▲ (ug/L)
Acrylic acid	SL-SOP	68	25-150	≤40	12	50
Asulam	SL-SOP	105	34-155	≤34	0.50	2.0
Cyanuric acid	SL-SOP	102	54-134	≤30	80	500
Ethylenethiourea	SL-SOP	104	60-111	≤36	1.2	5.0
Maleic acid/Maleic anhydride	SL-SOP	103	60-140	≤30	2.5	10
Nitrocellulose	SL-SOP	108	30-110	≤52	130	600
Phthalic acid/Phthalic anhydride	SL-SOP	103	66-127	≤30	1.0	10

TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/L)	RL^ (ug/L)
Dissolved Gases In Water (GC/FID)						
Methane	SL SOP	95	75-125	≤30	0.040	0.19
Ethane	SL SOP	95	75-125	≤30	0.050	0.35
Propane	SL SOP	95	75-125	≤30	0.13	0.51
Butane	SL SOP	95	75-125	≤30	0.17	0.68
Pentane	SL SOP	95	75-125	≤30	0.21	0.84
Hexane	SL SOP	95	75-125	≤30	0.25	1.0
Ethene	SL SOP	95	75-125	≤30	0.051	0.33
1-Propene	SL SOP	95	75-125	≤30	0.25	0.49
1-Butene	SL SOP	95	75-125	≤30	0.25	0.65
1-Pentene	SL SOP	95	75-125	≤30	0.25	0.82
1-Hexene	SL SOP	95	75-125	≤30	0.25	0.98
Dissolved Gases In Water (GC/TCD)						
Carbon dioxide	SL SOP	95	75-125	≤30	32	2600
Carbon monoxide	SL SOP	95	75-125	≤30	21	82
Ethane	SL SOP	95	75-125	≤30	12	88
Ethene	SL SOP	95	75-125	≤30	5.4	82
Methane	SL SOP	95	75-125	≤30	3.0	47
Nitrogen	SL SOP	95	75-125	≤30	200	200
Oxygen	SL SOP	95	75-125	≤30	200	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RL^ (mg/kg)
Aluminum (ICP)	6010(3050)	2	75-125	≤20	5.8	20
Antimony (ICP)	6010(3050)	2	75-125	≤20	0.50	2.0
Antimony (GFAA)	7041(3050)	2	80-120	≤20	0.11	1.0
Arsenic (ICP)	6010(3050)	2	75-125	≤20	0.45	1.0
Arsenic (GFAA)	7060(3050)	2	80-120	≤20	0.35	1.0
	7062(3050)	2	80-120	≤20	0.25	1.0
Barium (ICP)	6010(3050)	2	75-125	≤20	0.16	1.0
Beryllium (ICP)	6010(3050)	2	75-125	≤20	0.057	0.40
Boron(ICP)	6010(3050)	2	75-125	≤20	0.59	5.0
Cadmium (ICP)	6010(3050)	2	75-125	≤20	0.087	0.50
Cadmium (GFAA)	7131 (3050)	2	80-120	≤20	0.087	0.10
Calcium (ICP)	6010(3050)	2	75-125	≤20	20	50
Chromium (ICP)	6010(3050)	2	75-125	≤20	0.17	1.0
Chromium (GFAA)	7191(3050)	2	80-120	≤20	0.079	1.0
Chromium, hexavalent	7196 (3060)	2	80-120	≤30	0.30	0.40
Cobalt (ICP)	6010 (3050)	2	75-125	≤20	0.19	1.0
Copper (ICP)	6010 (3050)	2	75-125	≤20	0.72	2.0
Copper (GFAA)	7211(3050)	2	80-120	≤20	0.042	1.0
Iron (ICP)	6010 (3050)	2	75-125	≤20	4.5	5.0
Lead (ICP)	6010 (3050)	2	75-125	≤20	0.42	0.50
Lead (GFAA)	7421(3050)	2	80-120	≤20	0.36	0.50
Lithium (GFAA)	7430(3050)	2	70-130	≤20	0.19	1.0
Lithium (FLES)	SL SOP	100	70-130	≤20	0.026	0.20
Magnesium (ICP)	6010 (3050)	2	75-125	≤20	6.8	50
Manganese (ICP)	6010(3050)	2	75-125	≤20	0.21	1.0
Mercury (CVAA)	7471	2	80-120	≤20	0.0028	0.020
	3112B	4	70-130	≤20	0.0028	0.020
Molybdenum (ICP)	6010(3050)	2	75-125	≤20	0.14	1.0
Nickel (ICP)	6010(3050)	2	75-125	≤20	0.43	4.0
Potassium (ICP)	6010(3050)	2	75-125	≤20	16	100
Selenium (ICP)	6010(3050)	2	75-125	≤20	0.43	1.0
Selenium (GFAA)	7740(3050)	2	80-120	≤20	0.66	1.0
	7742(3050)	2	80-120	≤20	0.38	1.0
Silica (water soluble)	6010(ASTM 3987-85)	2	70-130	≤20	0.67	10

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Silver (ICP)	6010(3050)	2	75-125	≤20	0.19	1.0
Silver (GFAA)	7761 (3050)	2	80-120	≤20	0.0040	0.10
Sodium (ICP)	6010(3050)	2	75-125	≤20	49	50
Thallium (ICP)	6010(3050)	2	75-125	≤20	0.57	1.0
Thallium (GFAA)	7841 (3050)	2	80-120	≤20	0.20	1.0
Tin (ICP)	6010(3050)	2	75-125	≤20	4.0	5.0
Titanium(ICP)	6010(3050)	2	70-130	≤20	0.40	1.0
Vanadium (ICP)	6010 (3050)	2	75-125	≤20	0.11	1.0
Zinc (ICP)	6010 (3050)	2	75-125	≤20	0.98	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
FLAA = flame atomic adsorption spectrophotometer
FLES = flame emission spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RL▲ (mg/kg)
Ammonia (as N)	350.1(EPA-CE:3-140)	3/46	75-125	≤30	0.075	0.50
	350.3(EPA-CE)	3/46	75-125	≤30	0.055	0.50
BOD	EPA-CE:3-380	46	60-140	≤40	NA	200
BTU	D240-87	38	70-130	≤30	NA	200 BTU/lb
Carbon, total organic	EPA-CE [Walkley-Black]	46 /43	60-140	≤40	64	500
	9060	2	60-140	≤40	150	500
Cation exchange capacity	9080/EPA-CE:3-20	2/46	70-130	≤40	NA	0.0033 meq/100 g
	9081	2	70-130	≤40	NA	0.0033 meq/100 g
Chloride (extractable)(1)	9251	2	75-125	≤30	4.8	20
	9252	2	75-125	≤30	17	20
	4500-Cl⁻ C	4	75-125	≤30	5.0	20
	300.0/9056	82/2	75-125	≤25	0.68	20
Chloride, total	9251(5050)	2	70-130	≤40	NA	200
	9056(5050)	2	70-130	≤40	NA	200
COD	EPA-CE:3-373	46	60-140	≤40	NA	100
Coliform, fecal	9221C(EPA:62)	4/67	NA	≤200	NA	3 MPN/g
Coliform, total	9221B(EPA:62)	4/67	NA	≤200	NA	3 MPN/g
Cyanide, amenable to chlorination	9012(9013)	2	NA	≤50	NA	1.0
	9010(9013)	2	NA	≤40	NA	1.0
Cyanide, reactive	7.3.3.2/9014	2	NA	≤50	NA	100mg HCN/ Kg Waste
Cyanide, total	9012(9013)	2	75-125	≤30	0.50	1.0
	9012(9010)	2	75-125	≤30	0.50	1.0
Fluoride (extractable)(1)	340.2/4500F⁻C	3/4	75-125	≤25	0.88	4.0
	300.0/9056	82/2	75-125	≤25	0.20	2.0
Halogens, total	9056(5050)	2	70-130	≤40	NA	200
	9251(5050)	2	70-130	≤40	NA	200
Halogens, total organic (EOX)	EPA-600/4-84-008	2/44	60-140	≤50	8.6	10
Hydrogen ion (pH)(2)	9045	2	63-158	≤40	NA	NA
Ignitability	1010	2	NA	NA	NA	NA
Nitrate (as N)(extractable)	4500 NO_3^-F (EPA-CE:3-183)	4/46	75-125	≤30	1.0	5.0
	300.0/9056	82/2	75-125	≤25	0.18	5.0

[1]Extraction procedures for solids and semisolids are contained in Sections 8.2 and 8.3

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Nitrate-Nitrite(as N) (Extractable)	4500 NO_3^-F (EPA-CE:3-183)	4/46	75-125	≤30	1.0	5.0
Nitrite (as N)(Extractable)	4500 NO_3^-F (EPA-CE:3-183)	4/46	75-125	≤30	0.066	5.0
	300.0/9056	82/2	75-125	≤25	0.16	5.0
Nitrogen, organic	EPA-CE:3-205	46	NA	NA	NA	50
Nitrogen, total	TKN + NO_3/NO_2	46	NA	NA	NA	55
Nitrogen, total Kjeldahl	EPA-CE:3-201	46	65-135	≤50	17	50
Oil and Grease	9070(9071)	2	60-140	≤50	59	100
	413.2(9071)	3(2)	60-140	≤50	2.3	10
	1664 (HEM)	84	60-140	≤50	26	100
Orthophosphate (extractable)[1]	365.1/4500P F	3 / 4	75-125	≤30	0.18	5.0
Paint filter liquids	9095	2	NA	≤40	NA	NA
Perchlorate	300.0	113	75-125	≤25	0.0046	0.080
Petroleum hydrocarbons	418.1(3550)(sonication)	3/2	60-140	≤50	2.0	10
	418.1(3540)(soxhlet)	3/2	60-140	≤50	3.5	10
	1664 (HEM-SGT)	84	60-140	≤50	22	100
	5520F(5520D/E)	4	60-140	≤50	40	200
	9073(9071)	2	60-140	≤50	3.5	10
Phenolics, total recoverable	9065(EPA-CE:3-355)	2 (46)	60-140	≤40	0.76	1.0
Phosphorus, total	EPA-CE:3-213	46	60-140	≤40	6.8	25
	EPA-CE:3-212	46	60-140	≤40	6.3	25
Residue, fixed (% ash)	EPA-CE:3-59	46	NA	≤40	NA	0.10%
Solids, total	EPA-CE:3-58/2540G	46/4	NA	≤30	NA	0.10%
Solids, volatile	EPA-CE:3-59/2540G	46/4	75-125	≤30	NA	0.10%
Specific gravity	EPA-CE:3-61	46	NA	≤10	NA	NA
Streptococcus, fecal	9230B (EPA:III.D-135)	4/67	NA	NA	NA	3 MPN/g
Sulfate (extractable)	375.3	3	75-125	≤30	14	100
	375.4/9038	3/2	75-125	≤30	34	100
	300.0/9056	82/2	75-125	≤25	1.6	20

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Sulfide	9030/9034/ 4500S,E	2/4	50-150	≤50	2.0	25
Sulfide, acid volatile	SL-SOP	68	50-150	≤50	4.5	10
Sulfide, reactive	7.3.4.2/9030	2	NA	≤50	NA	50mg H_2S/ Kg Waste
Sulfur	D129-64/9056(5050)	38/2	70-130	≤30	NA	170:
Water (Karl Fisher)	E203-75	38	NA	≤30	NA	50
EP Toxicity	1310 Followed by 6010, 7470, 8080, 8150, 8240, and 8270	2	NA	NA	NA	NA
Synthetic Precipitation Leaching Procedure	1312 followed by requested analytical procedure(s): metals:6010 mercury: 7470 pesticides: 8081 herbicides: 8151 VOC:8260 BNA: 8270 cyanide: 9010/9012	2	Table 5.1	Table 5.1	Table 5.1	Table 5.1
Toxicity Characteristic Leaching Procedure	1311 followed by requested analytical procedure(s): metals: 6010 mercury: 7470 pesticides: 8081 herbicides: 8151 VOC: 8260 BNA:8270	2	Table 5.3	Table 5.3	Table 5.3	Table 5.3

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL A (ug/kg)
Acetone (MS)	8015(5035)	2	40-130	≤30	31	130
	8015(5035ext)	2	40-130	≤30	3100	5000
2-Butanone (MEK) (MS)	8015(5035)	2	60-130	≤40	20	130
	8015(5035ext)	2	60-130	≤40	2000	5000
Diethyl ether	8015(5035)	2	10-130	≤50	32	130
	8015(5035ext)	2	10-130	≤50	3200	5000
Heptane	8015(5035)	2	50-150	≤50	2.0	5.0
	8015(5035ext)	2	50-150	≤50	100	200
Hexane	8015(5035)	2	50-150	≤50	2.0	5.0
	8015(5035ext)	2	50-150	≤50	100	200
2-Hexanone	8015(5035)	2	50-150	≤40	50	130
	8015(5035ext)	2	50-150	≤40	2000	5000
4-Methyl-2-pentanone (MIBK) (MS)	8015(5035)	2	65-125	≤40	32	130
	8015(5035ext)	2	65-125	≤40	3200	5000
Methyl t-butyl ether (MTBE)	8015(5035)	2	50-150	≤30	23	50
	8015(5035ext)	2	50-150	≤30	1000	2000
Lacolene	8015(modified voaltiles) (5035)	2/12	60-140	≤30	100	250
	8015(modified volatiles) (5035ext)	2/12	60-140	≤30	4000	10000
Gasoline/GRO	GRO(Tennessee)	70	50-100	≤20	40	180
	GRO(ext)(Tennessee)	70	50-100	≤20	1600	7200
	GRO (8015 modified volatiles) (5035)	2	10-149	≤40	100	250
	GRO (8015 modified volatiles)(5035ext)	2	10-149	≤40	4000	10000
	8015 (modified volatiles) (5035)	2/12	10-149	≤40	100	250
	8015 (modified volatiles)(5035ext)	2/12	10-149	≤40	4000	10000
Volatile Petroleum Hydrocarbons (VPH)	TPH	107	70-130	≤25	540	5000
(Massachusetts's VPH Method)	C5-C8 Aliphatic Hydrocarbons	107	70-130	≤25	250	2000
	C9-C12 Aliphatic Hydrocarbons	107	70-130	≤25	320	500
	C9-C10 Aromatic Hydrocarbons	107	70-130	≤25	34	500
Surrogate - a,a,a-Trifluorotoluene	GRO(Tennessee)	70	50-150	NA	NA	NA
	8015/8015ext	2/12/70	41-156	NA	NA	NA
	Ma VPH	107	60-140	NA	NA	NA
Surrogate - 2,5-Dibromotoluene	Ma VPH	107	60-140	NA	NA	NA

(ext)= methanol extraction 1mL of methanol per gram of sample

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Petroleum hydrocarbons	FL-PRO(sonication)	83	26-116	≤25	4000	10000
	FL-PRO(Soxhlet)	83	41-224	≤25	4000	10000
	8015 (modified extractable) (3550)	2	40-140	≤40	4000	10000
Extractable Petroleum Hydrocarbons (EPH)	TPH	106	40-140	≤25	920	10000
(Massachusetts's EPH Method)	C9-C18 Aliphatic Hydrocarbons	106	40-140	≤25	330	10000
	C19-C36 Aliphatic Hydrocarbons	106	40-140	≤25	920	10000
	C11-C22 Aromatic Hydrocarbons	106	40-140	≤25	1600	10000
Texas TPH	TPH	114	70-130	≤30	16000	50000
	C6-C10 Hydrocarbons	114	70-130	≤30	16000	50000
	C10-C28 Hydrocarbons	114	70-130	≤30	8800	50000
Diesel/DRO	DRO	2/69	40-140	≤40	800	3300
	8015 (modified extractable) (3550)	2/12	10-127	≤40	2500	10000
Heavy oil	8015 (modified extractable) (3550)	2/12	40-140	≤40	1000	20000
Kerosene	8015 (modified extractable) (3550)	2/12	40-140	≤40	25000	10000
Mineral Spirits	8015 (modified extractable) (3550)	2/12	40-140	≤40	25000	10000
Surrogate - 2-Fluorobiphenyl	8015 (modified extractable) (3550)	2/12/69	10-152	NA	NA	NA
Surrogate - o-Terphenyl	DRO	2/69	15-154	NA	NA	NA
	8015 (modified extractable) (3550)	2/12	15-154	NA	NA	NA
	FL-PRO(sonication)	83	15-154	NA	NA	NA
	FL-PRO(Soxhlet)	83	57-115	NA	NA	NA
	EPH	106	40-140	NA	NA	NA
Surrogate-Nonatricontane (C39)	FL-PRO(sonication)	83	30-118	NA	NA	NA
	FL-PRO(Soxhlet)	83	61-153	NA	NA	NA
Chloro-octadecane	EPH	106	40-140	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
ACETATES						
n-Butyl acetate	8015 (DAI*)	2	50-150	≤50	350	5000
sec-Butyl acetate	8015 (DAI*)	2	50-150	≤50	290	5000
Cellosolve acetate	8015 (DAI*)	2	50-150	≤50	430	5000
Ethyl acetate (MS)	8015 (DAI*)	2	43-160	≤50	630	5000\
Isoamyl acetate	8015 (DAI*)	2	50-150	≤50	490	10000
Isobutyl acetate	8015 (DAI*)	2	67-166	≤50	490	5000
Isopropyl acetate	8015 (DAI*)	2	67-166	≤50	460	5000
Methyl acetate	8015 (DAI*)	2	46-170	≤50	810	5000
Phenyl mercuric acetate	8015 (DAI*)	2	30-130	≤50	1000	5000
n-Propyl acetate (MS)	8015 (DAI*)	2	24-158	≤50	460	5000
ALCOHOLS						
Tert-Amyl alcohol	8015 (DAI*)	2	49-181	≤50	530	1000
Sec-Butanol	8015 (DAI*)	2	37-182	≤50	390	1000
n-Butanol	8015 (DAI*)	2	50-150	≤50	700	1000
tert-Butanol	8015 (DAI*)	2	67-183	≤50	480	1000
Diacetone alcohol	8015 (DAI*)	2	50-150	≤50	680	5000
Ethanol (MS)	8015 (DAI*)	2	60-129	≤50	840	1000
Methanol (MS)	8015 (DAI*)	2	61-128	≤50	800	1000
n-Propanol	8015 (DAI*)	2	50-150	≤50	680	1000
Isopropanol (MS)	8015 (DAI*)	2	61-148	≤50	510	1000
Isobutanol	8015 (DAI*)	2	50-150	≤50	710	1000
CELLOSOLVES						
Butyl cellosolve	8015 (DAI*)	2	50-150	≤50	390	5000
Ethyl cellosolve	8015 (DAI*)	2	20-180	≤50	ND	20000
GLYCOLS						
Diethylene glycol	8015 (DAI*)	2	45-112	≤50	1000	5000
Ethylene glycol (MS)	8015 (DAI*)	2	35-132	≤50	1900	5000
Propylene glycol (MS)	8015 (DAI*)	2	60-126	≤50	590	5000
Tetraethylene glycol	8015 (DAI*)	2	50-150	≤50	4200	10000
Triethylene glycol	8015 (DAI*)	2	50-150	≤50	3300	5000
MISCELLANEOUS						
Cyclohexanone	8015 (DAI*)	2	50-150	≤50	670	5000
1,4-Dioxane	8015 (DAI*)	2	50-150	≤50	320	5000
Mesityl oxide	8015 (DAI*)	2	50-150	≤50	260	5000
2-Nitropropane	8015 (DAI*)	2	50-150	≤50	520	5000
Tetrahydrofuran (MS)	8015 (DAI*)	2	67-146	≤50	290	5000

(DAI) = Direct Aqueous (Extract) Injectio

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Benzene (MS)	8021(5035)	2	54-154	≤31	3.3	5.0
	8021 (5035ext)	2	54-154	≤31	130	200
Bromobenzene	8021(5035)	2	43-179	≤56	2.5	25
	8021 (5035ext)	2	43-179	≤56	100	1000
Bromochloromethane	8021(5035)	2	34-128	≤52	2.0	5.0
	8021 (5035ext)	2	34-128	≤52	80	200
Bromodichloromethane	8021(5035)	2	74-171	≤54	1.1	5.0
	8021 (5035ext)	2	74-171	≤54	44	200
Bromoform	8021(5035)	2	74-148	≤40	2.5	25
	8021 (5035ext)	2	74-148	≤40	100	1000
Bromomethane	8021(5035)	2	19-221	≤78	4.0	5.0
	8021 (5035ext)	2	19-221	≤78	160	200
n-Butylbenzene	8021(5035)	2	50-150	≤25	1.1	5.0
	8021(5035ext)	2	50-150	≤25	44	200
sec-Butylbenzene	8021(5035)	2	50-150	≤25	2.8	5.0
	8021 (5035ext)	2	50-150	≤25	110	200
tert-Butylbenzene	8021(5035)	2	49-188	≤48	1.2	5.0
	8021 (5035ext)	2	49-188	≤48	48	200
Carbon tetrachloride	8021(5035)	2	76-142	≤50	1.4	5.0
	8021 (5035ext)	2	76-142	≤50	76	200
Chlorobenzene (MS)	8021(5035)	2	56-122	≤25	0.74	5.0
	8021 (5035ext)	2	56-122	≤25	30	200
Chloroethane	8021(5035)	2	34-227	≤52	4.0	5.0
	8021 (5035ext)	2	34-227	≤52	160	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Chloroform	8021(5035)	2	63-181	≤36	2.4	5.0
	8021 (5035ext)	2	63-181	≤36	96	200
Chloromethane	8021(5035)	2	29-173	≤53	3.3	5.0
	8021 (5035ext)	2	29-173	≤53	130	200
2-Chlorotoluene	8021(5035)	2	70-140	≤27	1.2	25
	8021 (5035ext)	2	70-140	≤27	48	1000
4-Chlorotoluene	8021(5035)	2	77-136	≤27	0.90	25
	8021 (5035ext)	2	77-136	≤27	36	1000
Dibromochloromethane	8021(5035)	2	70-161	≤56	1.9	5.0
	8021 (5035ext)	2	70-161	≤56	76	200
1,2-Dibromo-3-chloropropane	8021(5035)	2	24-145	≤56	3.9	25
	8021 (5035ext)	2	24-145	≤56	160	1000
1,2-Dibromoethane (EDB)	8021(5035)	2	34-168	≤77	2.0	10
	8021 (5035ext)	2	34-168	≤77	80	400
Dibromomethane	8021(5035)	2	37-214	≤59	1.5	10
	8021 (5035ext)	2	37-214	≤59	60	400
1,2-Dichlorobenzene	8021(5035)	2	57-118	≤39	0.96	5.0
	8021 (5035ext)	2	57-118	≤39	38	200
1,3-Dichlorobenzene	8021(5035)	2	57-117	≤28	1.0	5.0
	8021 (5035ext)	2	57-117	≤28	40	200
1,4-Dichlorobenzene	8021(5035)	2	59-117	≤42	1.3	5.0
	8021 (5035ext)	2	59-117	≤42	52	500

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Dichlorodifluoromethane	8021(5035)	2	27-207	≤50	2.8	5.0
	8021 (5035ext)	2	27-207	≤50	110	200
1,1-Dichloroethane	8021(5035)	2	48-209	≤36	2.8	5.0
	8021 (5035ext)	2	48-209	≤36	110	200
1,2-Dichloroethane	8021(5035)	2	49-207	≤58	1.1	5.0
	8021 (5035ext)	2	49-207	≤58	44	200
1,1-Dichloroethene (MS)	8021(5035)	2	56-185	≤35	2.0	5.0
	8021 (5035ext)	2	56-185	≤35	80	200
cis-1,2-Dichloroethene	8021(5035)	2	40-138	≤39	1.6	5.0
	8021(5035ext)	2	40-138	≤39	64	200
trans-1,2-Dichloroethene	8021(5035)	2	64-139	≤43	1.8	5.0
	8021 (5035ext)	2	64-139	≤43	72	200
1,2-Dichloropropane	8021(5035)	2	62-178	≤45	0.59	5.0
	8021 (5035ext)	2	62-178	≤45	24	200
1,3-Dichloropropane	8021(5035)	2	53-150	≤57	1.6	5.0
	8021 (5035ext)	2	53-150	≤57	64	200
2,2-Dichloropropane	8021(5035)	2	40-138	≤39	1.3	5.0
	8021 (5035ext)	2	40-138	≤39	52	200
1,1-Dichloropropene	8021(5035)	2	42-141	≤50	1.2	5.0
	8021 (5035ext)	2	42-141	≤50	48	200
cis-1,3-Dichloropropene	8021(5035)	2	44-146	≤45	1.2	5.0
	8021 (5035ext)	2	44-146	≤45	48	200
trans-1,3-Dichloropropene	8021(5035)	2	30-152	≤55	1.5	5.0
	8021 (5035ext)	2	30-152	≤55	60	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Ethylbenzene	8021(5035)	2	68-118	≤29	1.6	5.0
	8021 (5035ext)	2	68-118	≤29	64	200
Hexachlorobutadiene	8021(5035)	2	52-145	≤41	2.0	5.0
	8021 (5035ext)	2	52-145	≤41	80	200
Isopropylbenzene	8021(5035)	2	50-150	≤27	1.3	5.0
	8021 (5035ext)	2	50-150	≤27	52	200
p-Isopropyltoluene	8021(5035)	2	50-150	≤25	1.1	5.0
	8021 (5035ext)	2	50-150	≤25	44	200
Methylene chloride	8021(5035)	2	40-224	≤40	2.9	25
	8021 (5035ext)	2	40-224	≤40	76	1000
Methy t-butyl ether (MTBE)	8021(5035)	2	70-130	≤30	2.6	50
	8021 (5035ext)	2	70-130	≤30	100	2000
Naphthalene	8021(5035)	2	67-133	≤42	3.2	5.0
	8021(5035ext)	2	67-133	≤42	130	200
n-Propylbenzene	8021(5035)	2	50-150	≤25	1.3	5.0
	8021 (5035ext)	2	50-150	≤25	52	200
Styrene	8021(5035)	2	70-130	≤30	0.79	5.0
	8021 (5035ext)	2	70-130	≤30	32	200
1,1,1,2-Tetrachloroethane	8021(5035)	2	75-145	≤41	1.5	5.0
	8021 (5035ext)	2	75-145	≤41	60	200
1,1,2,2-Tetrachloroethane	8021(5035)	2	58-166	≤48	2.3	5.0
	8021 (5035ext)	2	58-166	≤48	92	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Tetrachloroethene	8021(5035)	2	53-150	≤57	0.49	5.0
	8021 (5035ext)	2	53-150	≤57	20	200
Toluene (MS)	8021(5035)	2	64-144	≤25	1.2	5.0
	8021 (5035ext)	2	64-144	≤25	48	200
1,2,3-Trichlorobenzene	8021(5035)	2	50-150	≤26	0.77	5.0
	8021 (5035ext)	2	50-150	≤26	31	200
1,2,4-Trichlorobenzene	8021(5035)	2	44-139	≤53	1.4	5.0
	8021 (5035ext)	2	44-139	≤53	56	200
1,1,1-Trichloroethane	8021(5035)	2	72-170	≤47	1.4	5.0
	8021 (5035ext)	2	72-170	≤47	56	200
1,1,2-Trichloroethane	8021(5035)	2	61-182	≤53	1.9	5.0
	8021 (5035ext)	2	61-182	≤53	76	200
Trichloroethene (MS)	8021(5035)	2	56-133	≤25	1.3	5.0
	8021 (5035ext)	2	56-133	≤25	52	200
Trichlorofluoromethane	8021(5035)	2	48-165	≤37	2.9	5.0
	8021 (5035ext)	2	48-165	≤37	120	200
1,2,3-Trichloropropane	8021(5035)	2	50-150	≤48	1.4	10
	8021 (5035ext)	2	50-150	≤48	56	400
1,2,4-Trimethylbenzene	8021(5035)	2	32-132	≤44	1.0	5.0
	8021(5035ext)	2	32-132	≤44	100	200
1,3,5-Trimethylbenzene	8021(5035)	2	50-150	≤25	0.99	5.0
	8021 (5035ext)	2	50-150	≤25	40	200
Vinyl Chloride	8021(5035)	2	20-216	≤61	3.3	5.0
	8021 (5035ext)	2	20-216	≤61	130	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
o-Xylene	8021(5035)	2	50-150	≤25	0.99	5.0
	8021 (5035ext)	2	50-150	≤25	40	200
m&p-Xylene	8021(5035)	2	62-138	≤49	1.9	5.0
	8021 (5035ext)	2	62-138	≤49	76	200
Total Xylenes	8021(5035)	2	62-138	≤49	1.9	10
	8021 (5035ext)	2	62-138	≤49	76	400
Surrogate* -2-Bromo-1-chloropropane	8021(5035)	2	70-130	NA	NA	NA
	8021 (5035ext)	2	70-130	NA	NA	NA
Surrogate* – Fluorobenzene	8021(5035)	2	43-137	NA	NA	NA
	8021 (5035ext)	2	43-137	NA	NA	NA
Surrogate* -1-Bromo-3-chloropropane	8021(5035)	2	43-137	NA	NA	NA
	8021 (5035ext)	2	43-137	NA	NA	NA
Surrogate*- Bromochloromethane	8021(5035)	2	43-127	NA	NA	NA
	8021 (5035ext)	2	43-127	NA	NA	NA
Surrogate*- a,a,a-Trifluorotoluene	8021(5035)	2	41-156	NA	NA	NA
	8021 (5035ext)	2	41-156	NA	NA	NA
Surrogate*- 1,4-Dichlorobutane	8021(5035)	2	70-130	NA	NA	NA
	8021 (5035ext)	2	70-130	NA	NA	NA
Surrogate*- Bromochlorobenzene	8021(5035)	2	70-130	NA	NA	NA
	8021 (5035ext)	2	70-130	NA	NA	NA

(ext)= methanol extraction; 1ml methanol per gram of sample
*only one surrogate required per detector

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	RE	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2-Chlorophenol (MS)	8041(3550)	2	27-150	≤26	186	330
4-Chloro-3-methylphenol (MS)	8041(3550)	2	10-145	≤39	146	330
2,4-Dichlorophenol	8041(3550)	2	10-159	≤40	159	330
2,6-Dichlorophenol	8041(3550)	2	38-129	≤25	64	330
2,4-Dimethylphenol	8041(3550)	2	24-118	≤40	131	330
2,4-Dinitrophenol	8041(3550)	2	12-145	≤65	143	1700
2-Methyl-4,6-dinitrophenol	8041(3550)	2	30-136	≤40	154	1700
3 and 4-Methyl phenol (m & p cresol)	8041(3550)	2	10-150	≤50	180	330
2-Methyl phenol (o-cresol)	8041(3550)	2	10-150	≤50	92	330
Cresols (total)	8040(3550)	2	10-150	≤50	82	330
2-Nitrophenol	8041(3550)	2	43-117	≤40	127	330
4-Nitrophenol (MS)	8041(3550)	2	10-183	≤34	218	1700
Pentachlorophenol (MS)	8041(3550)	2	10-234	≤80	225	1700
Phenol (MS)	8041(3550)	2	10-138	≤30	146	330
2,3,4,6-Tetrachlorophenol	8041(3550)	2	50-150	≤40	160	330
2,3,4,5-Tetrachlorophenol	8041(3550)	2	50-150	≤40	120	330
Tetrachlorophenols (2,3,4,5 – 2,3,4,6)	8040(3550)	2	NA	NA	160	330
2,4,6-Trichlorophenol	8041(3550)	2	53-119	≤40	201	330
2,4,5-Trichlorophenol	8041(3550)	2	53-119	≤40	100	330
Trichlorophenols (2,4,5 + 2,4,6)	8041(3550)	2	NA	NA	100	330
Surrogate - 2,4,6-Tribromophenol	8041(3550)	2	10-138	NA	NA	NA
Surrogate - 2-Fluorophenol	8041(3550)	2	10-138	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Butyl benzyl phthalate (MS)	8061(3550)	2	36-209	≤66	28	330
Bis(2-ethylhexyl) phthalate (MS)	8061(3550)	2	39-195	≤54	34	330
Di-n-butyl phthalate (MS)	8061(3550)	2	34-195	≤41	28	330
Diethyl phthalate (MS)	8061(3550)	2	43-154	≤34	21	330
Dimethyl phthalate (MS)	8061(3550)	2	37-147	≤31	23	330
Di-n-octyl phthalate (MS)	8061(3550)	2	37-147	≤86	35	330
Surrogate-2-Flurobiphenyl	8061(3550)	2	10-150	NA	NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m- xylene (TCMX)	8061(3550)	2	30-150	NA	NA	NA
Surrogate- Decachlorobiphenyl (DCB)	8061(3550)	2	30-150	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
N-Nitrosodimethylamine	8070(3550)	2	13-109	≤50	12	67
N-Nitrosodi-n-propylamine	8070(3550)	2	45-146	≤50	6.8	67
N-Nitrosodiphenylamine	8070(3550)	2	10-139	≤50	5.7	100
Surrogate - Triphenylphosphate	8070(3550)	2	10-137	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Aldrin (MS)	8081(3550)	2	10-144	≤38	0.15	1.7
Benfluralin	8081(3550)	2	40-140	≤40	0.33	0.33
alpha-BHC	8081(3550)	2	22-101	≤40	0.13	1.7
beta-BHC	8081(3550)	2	12-120	≤40	0.62	1.7
Gamma-BHC (Lindane) (MS)	8081(3550)	2	12-138	≤37	0.57	1.7
delta-BHC	8081(3550)	2	10-142	≤47	0.083	1.7
Captafol	8081(3550)	2	20-180	≤50	0.83	3.3
Captan	8081(3550)	2	40-150	≤40	0.25	1.7
Technical Chlordane	8081(3550)	2	45-119	≤40	3.4	17
alpha Chlordane	8081(3550)	2	45-140	≤40	0.23	1.7
Gamma Chlordane	8081(3550)	2	11-141	≤40	0.20	1.7
Chlorobenzilate	8081(3550)	2	50-150	≤40	2.3	17
Chloroneb	8081(3550)	2	49-125	≤30	3.2	13
Chloropropylate	8081(3550)	2	51-125	≤30	4.0	16
Chlorothalonil	8081(3550)	2	35-130	≤40	1.7	6.7
Dacthal (DCPA)	8081(3550)	2	75-127	≤27	0.024	1.7
4,4'-DDD	8081(3550)	2	28-134	≤50	0.35	3.3
4,4'-DDE	8081(3550)	2	34-121	≤25	0.24	3.3
4,4'-DDT (MS)	8081(3550)	2	29-134	≤26	0.35	3.3
Dicofol (Kelthane)	8081(3550)	2	40-125	≤40	1.7	20
Dieldrin (MS)	8081(3550)	2	28-137	≤30	0.24	3.3
Endosulfan I	8081(3550)	2	10-141	≤40	0.14	1.7
Endosulfan II	8081(3550)	2	10-159	≤65	0.43	3.3
Endosulfan sulfate	8081(3550)	2	26-144	≤50	0.44	3.3
Endrin (MS)	8081(3550)	2	33-149	≤32	0.42	3.3
Endrin aldehyde	8081(3550)	2	10-130	≤86	0.24	3.3
Endrin ketone	8081(3550)	2	29-112	≤31	0.65	3.3
Etridiazole	8081(3550)	2	50-125	≤30	0.33	0.33
Heptachlor (MS)	8081(3550)	2	17-138	≤38	0.20	1.7
Heptachlor epoxide	8081(3550)	2	15-142	≤40	0.12	1.7
Isodrin	8081(3550)	2	10-150	≤50	0.36	3.3
Kepone(1)	8081(3550)	2	10-150	≤50	0.85	170
Methoxychlor	8081(3550)	2	24-152	≤40	0.51	17
Mirex	8081(3550)	2	20-100	≤50	1.6	33
Pentachloronitrobenzene (PCNB)	8081(3550)	2	40-150	≤40	0.13	3.3

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Pendimethalin	8081(3550)	2	35-125	≤50	17	67
Permethrin (total)	8081(3550)	2	40-140	≤50	8.2	33
Propachlor	8081(3550)	2	51-125	≤30	2.5	16
Toxaphene	8081(3550)	2	41-126	≤50	33	170
Trifluralin	8081(3550)	2	40-140	≤40	0.20	0.66
PCB-1016	8082(3550)	2	34-138	≤44	5.7	33
PCB 1221	8082(3550)	2	15-178	≤30	7.6	67
PCB 1232	8082(3550)	2	10-215	≤30	3.5	33
PCB-1242	8082(3550)	2	39-150	≤30	6.5	33
PCB-1248	8082(3550)	2	38-158	≤30	5.9	33
PCB-1254	8082(3550)	2	40-122	≤30	8.2	33
PCB-1260	8082(3550)	2	39-138	≤30	8.0	33
PCB-1268	8082(3550)	2	40-140	≤30	4.0	33
Surrogate - Dibutylchlorendate (DBC)	8081(3550)	2	30-150	NA	NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081/8082(3550)	2	30-150	NA	NA	NA
Surrogate- Decachlorobiphenyl (DCB)	8081/8082(3550)	2	30-150	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Individual Congeners (*)						
2,4'-Dichlorobiphenyl (8)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',5-Trichlorobiphenyl (18)	8082(3550)	2	50-150	≤50	0.25	1.0
2,4,4'-Trichlorobiphenyl (28)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,5'-Tetrachlorobiphenyl (44)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',5,5'-Tetrachlorobiphenyl (52)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3',4,4'-Tetrachlorobiphenyl (66)	8082(3550)	2	50-150	≤50	0.25	1.0
3,3',4,4'-Tetrachlorobiphenyl (77)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',4,5,5'-Pentachlorobiphenyl (101)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3,3',4,4'-Pentachlorobiphenyl (105)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',4,5,5'-Pentachlorobiphenyl (101)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3',4,4',5-Pentachlorobiphenyl (118)	8082(3550)	2	50-150	≤50	0.25	1.0
3,3',4,4',5-Pentachlorobiphenyl (126)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,3',4,4'-Hexachlorobiphenyl (128)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,4,4',5'-Hexachlorobiphenyl (138)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',4,4',5,5'-Hexachlorobiphenyl (153)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3,3',4,4',5-Hexachlorobiphenyl (156)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3,3',4,4',6-Hexachlorobiphenyl (158)	8082(3550)	2	50-150	≤50	0.25	1.0
2,3',4,4',5,5'-Hexachlorobiphenyl (167)	8082(3550)	2	50-150	≤50	0.25	1.0
3,3',4,4',5,5'-Hexachlorobiphenyl (169)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,3',4,4',5-Heptachlorobiphenyl (170)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,4,4',5,5'-Heptachlorobiphenyl (180)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,4',5,5',6-Heptachlorobiphenyl (187)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,3',4,4',5,6-Octachlorobiphenyl (195)	8082(3550)	2	50-150	≤50	0.25	1.0
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (206)	8082(3550)	2	50-150	≤50	0.25	1.0
Decachlorobiphenyl (209)	8082(3550)	2	50-150	≤50	0.25	1.0
Surrogate - 2,4,5,6-Tetrachloro-m- xylene (TCMX)	8082(3550)	2	30-150	NA	NA	NA
Surrogate- Decachlorobiphenyl (DCB)	8082(3550)	2	30-150	NA	NA	NA
Surrogate- Octachloronaphthalene	8082(3550)	2	30-150	NA	NA	NA

*These congeners are representitive of the 209 individual PCBs that may be determined using Method 8082. The number in parenthesis after the compound name is the PCB congener number. PCBs as Aroclors are listed in the previous section.

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Monochlorobiphenyls	680/(3550)	93	30-130	≤50	0.68	3.3
Dihlorobiphenyls	680/(3550)	93	30-130	≤50	0.76	3.3
Trichlorobiphenyls	680/(3550)	93	30-130	≤50	0.68	3.3
Tetrachlorobiphenyls	680/(3550)	93	40-140	≤50	1.3	6.7
Pentachlorobiphenyls	680/(3550)	93	40-140	≤50	0.83	6.7
Hexachlorobiphenyls	680/(3550)	93	40-140	≤50	0.89	6.7
Heptachlorobiphenyls	680/(3550)	93	40-140	≤50	1.6	10
Octachlorobiphenyls	680/(3550)	93	40-140	≤50	0.95	10
Nonachlorobiphenyls	680/(3550)	93	30-130	≤50	1.9	17
Decachlorobiphenyl	680/(3550)	93	30-130	≤50	1.9	17
Surrogate- Decachlorobiphenyl-13C12	680/(3550)	93	30-130	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,4-Dinitrotoluene (MS)	8091(3550)(ECD)	2	10-125	≤40	0.56	10
2,6-Dinitrotoluene (MS)	8091(3550)(ECD)	2	10-126	≤40	0.65	10
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8091(3550)(ECD)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl	8091(3550)(ECD)	2	30-150	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Anthracene*1	8100(3550)	2	38-118	≤32	28	330
Acenaphthene (MS)	8100(3550)	2	24-114	≤32	12	330
Acenaphthylene	8100(3550)	2	36-114	≤32	11	330
Benzo(a)anthracene*2	8100(3550)	2	30-127	≤42	35	330
Benzo(a)pyrene (MS)	8100(3550)	2	35-136	≤45	36	330
Benzo(b)fluoranthene*3	8100(3550)	2	26-128	≤41	24	330
Benzo(k)fluoranthene*3	8100(3550)	2	26-128	≤41	24	330
Benzo(g,h,i)perylene	8100(3550)	2	25-126	≤42	24	330
Carbazole	8100(3550)	2	40-140	≤40	42	330
Chrysene*2	8100(3550)	2	30-127	≤42	35	330
Dibenzo(a,h)anthracene*4	8100(3550)	2	20-131	≤47	47	330
Fluoranthene	8100(3550)	2	28-132	≤33	28	330
Fluorene (MS)	8100(3550)	2	32-114	≤33	9.7	330
Indeno(1,2,3-cd)Pyrene*4	8100(3550)	2	20-131	≤47	47	330
1-Methylnaphthalene	8100(3550)	2	20-140	≤50	18	330
2-Methylnaphthalene	8100(3550)	2	20-140	≤50	18	330
Napthalene (MS)	8100(3550)	2	10-133	≤45	19	330
Phenanthrene*1	8100(3550)	2	38-118	≤32	28	330
Pyrene (MS)	8100(3550)	2	39-121	≤32	14	330
Surrogate - 2-Fluorobiphenyl	8100(3550)	2	10-152	NA	NA	NA
Surrogate - o-Terphenyl	8100	12	15-154	NA	NA	NA

*# = where the number is the same for any 2 compounds, these compounds cannot be routinely resolved hromatographically and are therfore reported as a combined result.

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Bis(2-chloroethoxy)methane	8110(3550)	2	12-128	≤50	79	170
Bis(2-chloroethyl)ether	8110(3550)	2	11-152	≤50	120	670
Bis(2-chloroisopropyl)ether	8110(3550)	2	9-165	≤50	58	330
4-Bromophenyl phenyl ether	8110(3550)	2	D-189	≤50	46	170
4-Chlorophenyl phenyl ether	8110(3550)	2	D-170	≤50	500	1300
Surrogate - 2,4,5,6-Tetrachloro-m-xylene	8110(3550)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl	8110(3550)	2	30-150	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2-Chloronaphthalene	8121(3550)	2	9-148	≤50	42	330
1,2-Dichlorobenzene	8121(3550)	2	9-160	≤50	28	330
1,3-Dichlorobenzene	8121(3550)	2	D-150	≤50	34	330
1,4-Dichlorobenzene	8121(3550)	2	13-137	≤50	120	330
Hexachlorobenzene	8121(3550)	2	15-159	≤50	0.12	3.3
Hexachlorobutadiene	8121(3550)	2	D-139	≤50	0.17	3.3
Hexachlorocylcopentadiene	8121(3550)	2	D-111	≤50	0.80	3.3
Hexachloroethane	8121(3550)	2	8-139	≤50	0.11	3.3
1,2,4-Trichlorobenzene	8121(3550)	2	50-150	≤50	5.8	3.3
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8121(3550	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	8121(3550)	2	30-150	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acephate(1)	1657	72	40-140	≤50	42	167
Methamidophos(1)	1657	72	18-98	≤33	15	66
Alachlor	8141(3550)	2	40-140	≤30	4.6	33
Ametryn	8141(3550)	2	40-140	≤50	8.5	66
Atrazine	8141(3550)	2	57-123	≤39	11	66
Azinphos methyl	8141(3550)	2	16-129	≤50	7.0	66
Benoxacor	8141(3550)	2	48-113	≤46	132	330
Bolstar (Sulprofos)	8141(3550)	2	58-156	≤40	5.5	33
Bromacil	8141(3550)	2	40-140	≤50	6.1	66
Butylate	8141(3550)	2	38-145	≤76	73	66
Carbophenothion (Trithion)	8141(3550)	2	11-175	≤40	6.2	66
Chlorpyrifos	8141(3550)	2	22-110	≤40	4.3	33
Chlordimeform	8141(3550)	2	DL-121	≤67	115	330
5-Chloroaminotoluene	8141(3550)	2	37-106	≤42	132	330
Cyanazine	8141(3550)	2	40-150	≤23	22	33
Coumaphos	8141(3550)	2	51-147	≤40	8.5	330
Cycloate	8141(3550)	2	46-159	≤47	28	66
Demeton-O	8141(3550)	2	36-120	≤40	4.8	83
Demeton-S	8141(3550)	2	17-131	≤40	4.0	83
Diazinon (MS)	8141(3550)	2	41-128	≤30	7.0	33
Dichlofenthion	8141(3550)	2	10-132	≤50	6.9	33
Dichlorvos	8141(3550)	2	10-130	≤40	1.3	66
Dimethoate	8141(3550)	2	38-120	≤40	15	66
Disulfoton	8141(3550)	2	10-134	≤93	2.6	66
Dioxathion	8141(3550)	2	40-140	≤50	38	330
EPN	8141(3550)	2	48-124	≤30	11	33
EPTC	8141(3550)	2	46-154	≤55	32	66
Ethion	8141(3550)	2	10-200	≤40	3.2	17
Ethoprop	8141(3550)	2	58-113	≤40	9.7	17
Famphur	8141(3550)	2	10-129	≤60	4.3	66
Fenamiphos	8141(3550)	2	40-160	≤40	20	17
Fensulfothion	8141(3550)	2	43-145	≤40	6.3	330
Fenthion	8141(3550)	2	10-128	≤60	2.4	33
Fonophos	8141(3550)	2	40-160	≤40	8.2	33
Hexazinone	8141(3550)	2	40-140	≤50	14	33
Isofenphos	8141(3550)	2	40-160	≤40	3.2	17
Malathion	8141(3550)	2	10-141	≤40	7.6	33

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Merphos	8141(3550)	2	30-147	≤40	10	33
Metalaxyl	8141(3550)	2	40-140	≤50	8.2	33
Methyl chlorpyrifos	8141(3550)	2	10-164	≤50	6.3	33
Metolachlor	8141(3550)	2	28-130	≤54	5.5	33
Metribuzin	8141(3550)	2	40-140	≤50	5.5	33
Mevinphos	8141(3550)	2	30-100	≤40	3.8	66
Molinate	8141(3550)	2	37-127	≤74	26	66
Monocrotophos	8141(3550)	2	40-140	≤50	130	330
Naled	8141(3550)	2	54-130	≤40	6.9	330
Norflurazon	8141(3550)	2	40-140	≤50	14	33
Parathion, ethyl (MS)	8141(3550)	2	24-151	≤79	6.8	33
Parathion, methyl (MS)	8141(3550)	2	36-149	≤40	4.2	17
Pebulate	8141(3550)	2	22-172	≤50	30	33
Phorate	8141(3550)	2	36-125	≤40	2.4	33
Prometon	8141(3550)	2	40-140	≤50	5.9	66
Prometryn	8141(3550)	2	40-140	≤50	31	66
Propazine	8141(3550)	2	40-140	≤55	24	66
Ronnel (MS)	8141(3550)	2	26-130	≤35	4.0	33
Simazine	8141(3550)	2	10-181	≤59	16	66
Stirophos (Tetrachlorvinphos)	8141(3550)	2	36-126	≤40	5.4	33
Sulfotepp (MS)	8141(3550)	2	13-171	≤65	7.7	17
Terbufos	8141(3550)	2	40-140	≤50	8.4	17
Terbutryn	8141(3550)	2	40-140	≤50	82	330
Terbutylazine	8141(3550)	2	40-140	≤50	58	66
Thionazin (MS)	8141(3550)	2	10-116	≤60	2.9	33
Tokuthion (Prothiofos)	8141(3550)	2	14-128	≤40	9.1	33
Triadimefon	8141(3550)	2	40-140	≤50	7.2	33
Trichloronate	8141(3550)	2	49-161	≤40	3.6	330
Tris-(2,3-Dibromopropyl) phosphate	8141(3550)	2	57-159	≤48	730	1000
Vernolate	8141(3550)	2	39-147	≤45	11	66
Surrogate - Triphenylphosphate	8141(3550)	2	30-137	NA	NA	NA

(1) Determined by NPD.

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Bentazon	8151	2	30-130	≤48	11	35
2,4-D (MS)	8151	2	19-153	≤47	1.6	8.3
Dalapon	8151	2	10-170	≤40	23	2000
2,4-DB	8151	2	20-160	≤40	6.4	8.3
Dicamba	8151	2	20-160	≤40	1.2	20
Dichlorprop	8151	2	30-170	≤40	2.7	100
Dinoseb	8151	2	10-130	≤50	8.3	100
MCPA	8151	2	10-130	≤50	740	2000
MCPP	8151	2	10-130	≤50	400	2000
Pentachlorophenol	8151	2	10-150	≤40	0.96	17
Picloram	8151	2	10-150	≤40	1.2	3.3
2,4,5-T (MS)	8151	2	14-143	≤59	1.0	8.3
2,4,5-TP (Silvex) (MS)	8151	2	27-120	≤51	0.90	8.3
Surrogate - 2,4-Dichlorophenoxy butanoic acid (2,4-DB)	8151	2	30-160	NA	NA	NA
Surrogate - 2,4-Dichlorophenyl acetic acid (DCAA)	8151	2	30-189	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acetone	8260(5035)	2	43-154	≤28	3.6	50
	8260(5035ext)	2	43-154	≤28	140	2000
Acetonitrile	8260(5035)	2	78-151	≤40	62	200
	8260(5035ext)	2	78-151	≤40	6200	8000
Acrolein	8260(5035)	2	22-164	≤65	38	100
	8260(5035ext)	2	22-164	≤65	1500	4000
Acrylonitrile	8260(5035)	2	41-140	≤40	11	100
	8260(5035ext)	2	41-140	≤40	440	4000
Benzene (MS)	8260(5035)	2	49-142	≤42	1.7	5.0
	8260(5035ext)	2	49-142	≤42	68	200
Benzyl Chloride	8260(5035)	2	50-150	≤40	25	100
	8260(5035ext)	2	50-150	≤40	1000	4000
Bromobenzene	8260(5035)	2	34-145	≤33	1.2	5.0
	8260(5035ext)	2	34-145	≤33	48	200
Bromochloromethane	8260(5035)	2	41-146	≤30	1.2	5.0
	8260(5035ext)	2	41-146	≤40	48	200
Bromodichloromethane	8260(5035)	2	32-149	≤33	0.88	5.0
	8260(5035ext)	2	32-149	≤33	35	200
Bromoform	8260(5035)	2	41-138	≤24	0.82	5.0
	8260(5035ext)	2	41-138	≤24	33	200
Bromomethane	8260(5035)	2	23-173	≤79	3.6	10
	8260(5035ext)	2	23-173	≤79	140	400
2-Butanone (MEK)	8260(5035)	2	45-154	≤39	3.3	25
	8260(5035ext)	2	45-154	≤39	130	1000
n-Butylbenzene	8260(5035)	2	29-142	≤72	2.1	5.0
	8260(5035ext)	2	29-142	≤72	84	200
sec-Butylbenzene	8260(5035)	2	35-142	≤63	2.4	5.0
	8260(5035ext)	2	35-142	≤63	96	200
tert-Butylbenzene	8260(5035)	2	54-130	≤40	2.0	5.0
	8260(5035ext)	2	54-130	≤40	80	200
Carbon disulfide	8260(5035)	2	40-135	≤68	2.2	5.0
	8260(5035ext)	2	40-135	≤68	88	200
Carbon tetrachloride	8260(5035)	2	40-135	≤59	1.9	5.0
	8260(5035ext)	2	40-135	≤59	76	200
Chlorobenzene (MS)	8260(5035)	2	66-135	≤34	1.3	5.0
	8260(5035ext)	2	66-135	≤34	52	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
2-Chloro-1,3-butadiene (Chloroprene)	8260(5035)	2	28-256	≤65	1.7	5.0
	8260(5035ext)	2	28-256	≤65	68	200
Chloroethane	8260(5035)	2	30-135	≤51	2.2	10
	8260(5035ext)	2	30-135	≤51	88	400
2-Chloroethyl vinyl ether	8260(5035)	2	D-208	≤28	3.2	50
	8260(5035ext)	2	D-208	≤28	130	2000
Chloroform	8260(5035)	2	50-133	≤38	1.7	5.0
	8260(5035ext)	2	50-133	≤38	68	200
Chloromethane	8260(5035)	2	32-142	≤53	1.3	10
	8260(5035ext)	2	32-142	≤53	52	400
3-Chloropropene (Allyl chloride)	8260(5035)	2	50-150	≤40	0.26	5.0
	8260(5035ext)	2	50-150	≤40	10	200
2-Chlorotoluene	8260(5035)	2	34-150	≤33	1.0	5.0
	8260(5035ext)	2	34-150	≤33	40	200
4-Chlorotoluene	8260(5035)	2	34-139	≤37	1.2	5.0
	8260(5035ext)	2	34-139	≤37	48	200
Cyclohexanone	8260 (5035)	2	38-111	≤52	240	750
	8260**(5035ext)	2	38-111	≤52	9600	30000
Dibromochloromethane	8260(5035)	2	47-135	≤22	0.84	5.0
	8260(5035ext)	2	47-135	≤22	34	200
1,2-Dibromo-3-chloropropane (DBCP)	8260(5035)	2	28-148	≤36	0.61	10
	8260(5035ext)	2	28-148	≤36	24	400
1,2-Dibromoethane (EDB)	8260(5035)	2	29-161	≤40	0.86	5.0
	8260(5035ext)	2	29-161	≤40	34	200
Dibromomethane	8260(5035)	2	35-155	≤24	0.93	5.0
	8260(5035ext)	2	35-155	≤24	37	200
1,2-Dichlorobenzene	8260(5035)	2	37-139	≤28	1.1	5.0
	8260(5035ext)	2	37-139	≤28	44	200
1,3-Dichlorobenzene	8260(5035)	2	35-143	≤34	1.3	5.0
	8260(5035ext)	2	35-143	≤34	52	200
1,4-Dichlorobenzene	8260(5035)	2	34-138	≤36	1.4	5.0
	8260(5035)	2	34-138	≤36	56	200
trans-1,4-Dichloro-2-butene	8260(5035)	2	79-178	≤40	0.94	10
	8260(5035ext)	2	79-178	≤40	38	400
Dichlorodifluoromethane	8260(5035)	2	53-126	≤41	2.4	5.0
	8260(5035ext)	2	53-126	≤41	96	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
1,1-Dichloroethane	8260(5035)	2	51-129	≤38	1.6	5.0
	8260(5035ext)	2	51-129	≤38	64	200
1,2-Dichloroethane	8260(5035)	2	49-136	≤25	1.2	5.0
	8260(5035ext)	2	49-136	≤25	48	200
cis-1,2-Dichloroethene	8260(5035)	2	44-124	≤37	1.7	5.0
	8260(5035ext)	2	44-124	≤37	68	200
trans-1,2-Dichloroethene	8260(5035)	2	37-142	≤56	1.9	5.0
	8260(5035ext)	2	37-142	≤56	76	200
1,1-Dichloroethene (MS)	8260(5035)	2	40-164	≤46	2.2	5.0
	8260(5035ext)	2	40-146	≤46	88	200
1,2-Dichloropropane	8260(5035)	2	52-124	≤27	1.4	5.0
	8260(5035ext)	2	52-124	≤27	56	200
1,3-Dichloropropane	8260(5035)	2	44-136	≤24	1.6	5.0
	8260(5035ext)	2	44-136	≤24	64	200
2,2-Dichloropropane	8260(5035)	2	43-129	≤53	2.0	5.0
	8260(5035ext)	2	43-129	≤53	80	200
1,1-Dichloropropene	8260(5035)	2	34-134	≤58	2.0	5.0
	8260(5035ext)	2	34-134	≤58	80	200
cis-1,3-Dichloropropene	8260(5035)	2	40-133	≤34	0.99	5.0
	8260(5035ext)	2	40-133	≤34	40	200
trans-1,3-Dichloropropene	8260(5035)	2	45-131	≤50	2.1	5.0
	8260(5035ext)	2	45-131	≤50	84	200
Diethyl ether	8260(5035)	2	50-150	≤40	2.5	10
	8260(5035ext)	2	50-150	≤40	100	400
Ethanol	8240(5030)8260 (5035)	2	40-160	≤40	250	1000
	8260(5035ext)	2	40-160	≤40	10000	40000
Ethyl Acetate	8260(5035)	2	73-137	≤21	1.7	5.0
	8260(5035ext)	2	73-137	≤21	68	200
Ethylbenzene	8260(5035)	2	51-135	≤44	2.0	5.0
	8260(5035ext)	2	51-135	≤44	80	200
Ethyl methacrylate	8260(5035)	2	50-150	≤40	1.6	5.0
	8260(5035ext)	2	50-150	≤40	64	200
Ethylene oxide	8260(5035)	2	D-411	≤83	900	3500
	8260**(5035ext)	2	D-411	≤83	36000	140000
Hexachlorobutadiene	8260(5035)	2	30-140	≤40	1.4	5.0
	8260**(5035ext)	2	30-140	≤40	56	200
2-Hexanone	8260(5035)	2	45-127	≤32	4.8	25
	8260(5035ext)	2	45-127	≤32	190	1000

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Iodomethane	8260(5035)	2	50-150	≤40	1.9	5.0
	8260(5035ext)	2	50-150	≤40	76	200
Isobutyl alcohol	8260(5035)	2	63-173	≤40	100	200
	8260(5035ext)	2	63-173	≤40	4000	8000
Isopropylbenzene	8260(5035)	2	38-148	≤39	3.8	5.0
	8260(5035ext)	2	38-148	≤39	150	200
p-Isopropyltoluene	8260(5035)	2	70-130	≤30	1.4	5.0
	8260(5035ext)	2	70-130	≤30	56	200
Methacrylonitrile	8260(5035)	2	69-145	≤60	32	100
	8260(5035ext)	2	69-145	≤60	1300	4000
Methylene chloride	8260(5035)	2	44-142	≤32	1.4	5.0
	8260(5035ext)	2	44-142	≤32	56	200
Methyl methacrylate	8260(5035)	2	50-150	≤45	2.2	5.0
	8260(5035ext)	2	50-150	≤45	88	200
4-Methyl-2-pentanone (MIBK)	8260(5035)	2	34-159	≤37	4.9	25
	8260(5035ext)	2	34-159	≤37	200	1000
Methyl t-butyl ether (MTBE)	8260(5035)	2	67-128	≤20	1.2	50
	8260(5035ext)	2	67-128	≤20	48	2000
Naphthalene	8260(5035)	2	37-131	≤29	0.57	5.0
	8260(5035ext)	2	37-131	≤29	23	200
2-Nitropropane	8260(5035)	2	35-128	≤47	2.4	10
	8260(5035ext)	2	35-128	≤47	96	400
Pentachloroethane	8260(5035)	2	41-165	≤50	10	25
	8260(5035ext)	2	41-165	≤50	400	1000
Propionitrile (ethylcyanide)	8260(5035)	2	73-227	≤65	37	100
	8260(5035ext)	2	73-227	≤65	1500	4000
n-Propylbenzene	8260(5035)	2	31-148	≤32	1.7	5.0
	8260(5035ext)	2	31-148	≤32	68	200
Styrene	8260(5035)	2	43-140	≤45	1.8	5.0
	8260(5035ext)	2	43-140	≤45	72	200
1,1,1,2-Tetrachloroethane	8260(5035)	2	38-132	≤32	1.3	5.0
	8260(5035ext)	2	38-132	≤32	52	200
1,1,2,2-Tetrachloroethane	8260(5035)	2	49-144	≤28	1.0	5.0
	8260(5035ext)	2	49-144	≤28	40	200
Tetrachloroethene	8260(5035)	2	71-146	≤44	3.0	5.0
	8260(5035ext)	2	71-146	≤44	120	200
Toluene (MS)	8260(5035)	2	38-158	≤32	1.8	5.0
	8260(5035ext)	2	38-158	≤32	72	200

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
1,2,3-Trichlorobenzene	8260(5035)	2	24-138	≤26	0.96	5.0
	8260(5035ext)	2	24-138	≤26	38	200
1,2,4-Trichlorobenzene	8260(5035)	2	22-138	≤35	1.0	5.0
	8260(5035ext)	2	22-138	≤35	40	200
1,1,1-Trichloroethane	8260(5035)	2	41-134	≤54	2.1	5.0
	8260(5035ext)	2	41-134	≤54	84	200
1,1,2-Trichloroethane	8260(5035)	2	34-148	≤27	0.96	5.0
	8260(5035ext)	2	34-148	≤27	38	200
Trichloroethene (MS)	8260(5035)	2	51-146	≤34	2.1	5.0
	8260(5035ext)	2	51-146	≤34	84	200
Trichlorofluoromethane	8260(5035)	2	32-138	≤30	2.8	5.0
	8260(5035ext)	2	32-138	≤30	110	200
1,2,3-Trichloropropane	8260(5035)	2	30-167	≤25	1.0	5.0
	8260(5035ext)	2	30-167	≤25	40	200
1,1,2-Trichloro-1,2,2-trifluoroethane	8260(5035)	2	17-181	≤65	2.8	5.0
	8260(5035ext)	2	17-181	≤65	110	200
1,2,4-Trimethylbenzene	8260(5035)	2	41-146	≤23	1.0	5.0
	8260(5035ext)	2	41-146	≤23	40	200
1,3,5-Trimethylbenzene	8260(5035)	2	38-144	≤28	1.2	5.0
	8260(5035ext)	2	38-144	≤28	48	200
Vinyl acetate	8260(5035)	2	10-174	≤133	1.0	10
	8260(5035ext)	2	10-174	≤133	40	400
Vinyl chloride	8260(5035)	2	33-142	≤65	1.5	10
	8260(5035ext)	2	33-142	≤65	60	400
Xylenes (total)	8260(5035)	2	37-133	≤43	3.0	10
	8260(5035ext)	2	37-133	≤43	120	400
o-Xylene	8260(5035)	2	41-148	≤32	1.6	5.0
	8260(5035ext)	2	41-148	≤32	64	200
m+p-Xylene	8260(5035)	2	37-133	≤43	3.0	5.0
	8260(5035ext)	2	37-133	≤43	120	200
Surrogate - Toluene-d8	8260(5035)	2	64-136	NA	NA	NA
	8260(5035ext)	2	64-136	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5035)	2	63-135	NA	NA	NA
	8260(5035ext)	2	63-135	NA	NA	NA
Surrogate –Dibromofluoromethane	8260(5035)	2	58-142	NA	NA	NA
	8260(5035ext)	2	58-142	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	8260(5035)	2	70-130	NA	NA	NA
	8260(5035ext)	2	70-130	NA	NA	NA
Surrogate -1,2-Dichlorobenzene-d4	8260(5035)	2	70-130	NA	NA	NA
	8260(5035ext)	2	70-130	NA	NA	NA

(ext) = methanol extraction; 1mL methanol per gram of sample

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Acenaphthene (MS)	8270(3550)	2	18-123	≤49	29	330
	8270(3550)-Low Level	2	18-123	≤49	1.5	6.7
Acenaphthylene	8270(3550)	2	42-119	≤48	21	330
	8270(3550)-Low Level	2	42-119	≤48	1.2	6.7
Acetophenone	8270(3550)	2	10-150	≤50	41	330
2-Acetylaminofluorene	8270(3550)	2	25-150	≤50	49	330
Aldrin	8270(3550)	2	10-166	≤40	76	330
4-Aminobiphenyl	8270(3550)	2	10-150	≤50	59	330
Aniline	8270(3550)	2	10-150	≤50	23	330
Anthracene	8270(3550)	2	40-148	≤27	21	330
	8270(3550)-Low Level	2	40-148	≤27	1.2	6.7
Aramite	8270(3550)	2	40-150	≤50	36	330
Benzidine	8270(3550)	2	10-200	≤100	36	2700
Benzo(a)anthracene	8270(3550)	2	54-137	≤43	20	330
	8270(3550)-Low Level	2	54-137	≤43	1.5	6.7
Benzoic acid	8270(3550)	2	10-150	≤50	61	1700
Benzo(b)fluoranthene	8270(3550)	2	43-134	≤51	23	330
	8270(3550)-Low Level	2	43-134	≤51	1.5	6.7
Benzo(k)fluoranthene	8270(3550)	2	25-182	≤48	26	330
	8270(3550)-Low Level	2	25-182	≤48	1.5	6.7
Benzo(g,h,i)perylene	8270(3550)	2	10-148	≤50	26	330
	8270(3550)-Low Level	2	10-148	≤50	1.3	6.7
Benzo(a)pyrene	8270(3550)	2	41-142	≤55	29	330
	8270(3550)-Low Level	2	41-142	≤55	1.2	6.7
Benzyl alcohol	8270(3550)	2/6	10-150	≤50	30	330
Benzyl chloride	8270(3550)	2	10-150	≤50	82	330
alpha-BHC	8270(3550)	2	10-150	≤50	56	330
beta-BHC	8270(3550)	2	24-149	≤40	55	330
delta-BHC	8270(3550)	2	10-110	≤40	55	330
gamma-BHC	8270(3550)	2	10-150	≤50	62	330
Biphenyl (Diphenyl)	8270(3550)	2	31-150	≤57	197	660
Bis(2-chloroethoxy) methane	8270(3550)	2	34-108	≤52	29	330
Bis(2-chloroethyl) ether	8270(3550)	2	18-122	≤50	26	330
Bis(2-chloroisopropyl) ether	8270(3550)	2	10-135	≤28	33	330
Bis(2-ethylhexyl) phthalate	8270(3550)	2	47-143	≤22	45	330
4-Bromophenyl phenyl ether	8270(3550)	2	31-157	≤19	24	330
Butyl benzyl phthalate	8270(3550)	2	58-122	≤27	42	330
Carbazole	8270(3550)	2	10-158	≤50	22	330

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
4-Chloroaniline	8270(3550)	2	10-130	≤85	37	660
4-Chloro-3-methylphenol (MS)	8270(3550)	2	24-114	≤32	49	330
1-Chloronaphthalene	8270(3550)	2	10-150	≤50	82	330
2-Chloronaphthalene	8270(3550)	2	39-107	≤47	24	330
2-Chlorophenol (MS)	8270(3550)	2	15-111	≤38	32	330
4-Chlorophenylphenyl ether	8270(3550)	2	36-149	≤62	28	330
Chrysene	8270(3550)	2	56-133	≤41	21	330
	8270(3550)-Low Level	2	56-133	≤41	2.1	6.7
2-Methyl phenol (o-Cresol)	8270(3550)	2	33-108	≤53	32	330
3- Methyl phenol (m- Cresol)	8270(3550)	2	24-114	≤42	42	330
4-Methyl phenol (p-Cresol)	8270(3550)	2	24-114	≤42	42	330
3- and 4-Methyl phenol	8270(3550)	2	24-114	≤42	42	330
4,4'-DDD	8270(3550)	2	10-145	≤40	36	330
4,4'-DDE	8270(3550)	2	10-136	≤40	57	330
4,4'-DDT	8270(3550)	2	10-203	≤62	56	330
Diallate	8270(3550)	2	10-150	≤50	39	330
Dibenz(a,h)anthracene	8270(3550)	2	31-129	≤24	52	330
	8270(3550)-Low Level	2	31-129	≤24	1.4	6.7
Dibenzofuran	8270(3550)	2	36-132	≤42	30	330
Dibenzo(a,e)pyrene	8270(3550)	2	35-109	≤42	110	330
Di-n-butylphthalate	8270(3550)	2	42-161	≤59	28	330
1,2-Dichlorobenzene	8270(3550)	2	25-115	≤24	24	330
1,3-Dichlorobenzene	8270(3550)	2	26-108	≤28	31	330
1,4-Dichlorobenzene (MS)	8270(3550)	2	10-105	≤31	27	330
3,3'-Dichlorobenzidine	8270(3550)	2	10-115	≤39	150	660
2,4-Dichlorophenol	8270(3550)	2	32-130	≤60	31	330
2,6-Dichlorophenol	8270(3550)	2	10-150	≤50	37	330
Dieldrin	8270(3550)	2	29-136	≤40	46	330
Diethylphthalate	8270(3550)	2	31-130	≤40	32	330
Dimethoate	8270(3550)	2	10-150	≤50	100	330
p-(Dimethylamino)azobenzene	8270(3550)	2	10-150	≤50	20	330
7,12-Dimethylbenz(a)anthracene	8270(3550)	2	10-150	≤50	17	330
3,3'-Dimethylbenzidine	8270(3550)	2	10-200	≤100	91	1700
a,a-Dimethylphenethylamine	8270(3550)	2	10-150	≤50	17000	67000
2,4-Dimethylphenol	8270(3550)	2	33-84	≤54	28	330
Dimethylphthalate	8270(3550)	2	49-130	≤45	25	330
m-Dinitrobenzene	8270(3550)	2	10-150	≤50	27	330
p-Dinitrobenzene	8270(3550)	2	49-105	≤40	400	1700
4,6-Dinitro-2-methylphenol	8270(3550)	2	10-117	≤57	200	1700

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,4-Dinitrophenol	8270(3550)	2	10-125	≤84	150	1700
2,4-Dinitrotoluene (MS)	8270(3550)	2	11-120	≤37	40	330
2,6-Dinitrotoluene	8270(3550)	2	10-112	≤45	29	330
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270(3550)	2	10-150	≤50	30	330
Di-n-octylphthalate	8270(3550)	2	22-181	≤43	54	330
1,4-Dioxane	8270(3550)	2	10-150	≤50	15	330
Diphenylamine/ N-nitrosodiphenylamine	8270(3550)	2	51-132	≤44	25	330
Diphenylamine	8270(3550)	2	51-132	≤44	25	330
1,2-Diphenyl hydrazine	8270(3550)	2	10-150	≤50	27	330
Disulfoton	8270(3550)	2	10-150	≤50	100	330
Endosulfan I	8270(3550)	2	10-150	≤50	80	660
Endosulfan II	8270(3550)	2	10-150	≤50	50	660
Endosulfan sulfate	8270(3550)	2	10-107	≤50	46	660
Endrin	8270(3550)	2	10-150	≤50	70	660
Endrin aldehyde	8270(3550)	2	10-209	≤50	72	1700
Endrin ketone	8270/(3550)	2	10-150	≤50	420	1700
Ethyl carbamate	8270(3550)	2	48-100	≤20	34	330
Ethyl methanesulfonate	8270(3550)	2	10-150	≤50	55	330
Ethyl parathion	8270(3550)	2	10-150	≤50	100	330
Famphur	8270(3550)	2	10-150	≤50	100	330
Fluoranthene	8270(3550)	2	39-157	≤50	31	330
	8270(3550)-Low Level	2	39-157	≤50	1.5	6.7
Fluorene	8270(3550)	2	27-151	≤50	31	330
	8270(3550)-Low Level	2	27-151	≤50	1.1	6.7
Heptachlor	8270(3550)	2	10-192	≤40	45	660
Heptachlor epoxide	8270(3550)	2	26-155	≤55	52	660
Hexachlorobenzene	8270(3550)	2	19-155	≤33	45	330
Hexachlorobutadiene	8270(3550)	2	33-114	≤55	23	330
Hexachlorocyclopentadiene	8270(3550)	2	D-132	≤50	51	330
Hexachloroethane	8270(3550)	2	10-109	≤30	24	330
Hexachlorophene[4]	8270(3550)	2	10-200	≤80	1400	170000
Hexachloropropene	8270(3550)	2	10-150	≤50	41	330
Indeno(1,2,3-cd)pyrene	8270(3550)	2	24-136	≤28	67	330
	8270(3550)-Low Level	2	24-136	≤28	1.0	6.7
Isophorone	8270(3550)	2	15-115	≤50	26	330
Isosafrole	8270(3550)	2	10-150	≤50	47	330
Kepone	8270(3550)	2	10-150	≤50	80	330
Methapyrilene	8270(3550)	2	10-150	≤50	960	67000
3-Methylcholanthrene	8270(3550)	2	10-150	≤50	47	330

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
4,4-Methylenebis(2-chloroaniline)	8270(3550)	2	40-122	≤57	4500	17000
Methyl methanesulfonate	8270(3550)	2	10-150	≤50	38	330
1-Methylnaphthalene	8270(3550)	2	10-150	≤50	30	330
	8270(3550)-Low Level	2	10-150	≤50	1.2	6.7
2-Methylnaphthalene	8270(3550)	2	30-133	≤63	32	330
	8270(3550)-Low Level	2	30-133	≤63	0.74	6.7
Methyl parathion	8270(3550)	2	10-150	≤50	100	330
Naphthalene	8270(3550)	2	25-131	≤34	25	330
	8270(3550)-Low Level	2	25-131	≤34	1.0	6.7
1,4-naphthoquinone	8270(3550)	2	10-150	≤50	27	330
1-Naphthylamine	8270(3550)	2	10-150	≤50	78	330
2-Naphthylamine	8270(3550)	2	10-150	≤50	77	330
Nicotine	8270(3550)	2	10-150	≤50	820	3300
2-Nitroaniline	8270(3550)	2	17-130	≤48	42	1700
3-Nitroanline	8270(3550)	2	14-130	≤28	32	1700
4-Nitroaniline	8270(3550)	2	10-130	≤55	42	1700
Nitrobenzene	8270(3550)	2	19-120	≤30	27	330
2-Nitrophenol	8270(3550)	2	30-130	≤50	19	330
4-Nitrophenol(MS)	8270(3550)	2	15-118	≤57	160	1700
4-Nitroquinoline-1-oxide	8270(3550)	2	10-150	≤50	190	3300
N-Nitroso-di-N-butylamine	8270(3550)	2	10-150	≤50	40	330
N-Nitrosodiethylamine	8270(3550)	2	10-150	≤50	18	330
N-Nitrosodimethylamine	8270(3550)	2	10-150	≤50	49	330
N-Nitrosodiphenylamine/ Diphenylamine	8270(3550)	2	51-132	≤44	25	330
N-Nitrosodiphenylamine	8270(3550)	2	51-132	≤44	25	330
N-Nitrosos-di-N-propylamine (MS)	8270(3550)	2	11-122	≤37	32	330
N-Nitrosomethylethylamine	8270(3550)	2	10-150	≤50	130	330
N-Nitrosomorpholine	8270(3550)	2	10-150	≤50	36	330
N-Nitrosopiperidine	8270(3550)	2	10-150	≤50	42	330
N-Nitrosopyrrolidine	8270(3550)	2	10-150	≤50	44	330
5-Nitro-o-toluidine	8270(3550)	2	10-150	≤50	21	330
Pentachlorobenzene	8270(3550)	2	10-150	≤50	49	330
Pentachloronitrobenzene	8270(3550)	2	10-150	≤50	26	330
Pentachlorophenol (MS)	8270(3550)	2	10-140	≤55	180	1700
Phenacetin	8270(3550)	2	10-150	≤50	55	330
Phenanthrene	8270(3550)	2	39-152	≤30	17	330
	8270(3550)-Low Level	2	39-152	≤30	1.2	6.7
Phenol (MS)	8270(3550)	2	13-115	≤39	42	330
Phenyl ether (Diphenyl oxide)	8270(3550)	2	29-157	≤61	212	660

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
p-Phenylenediamine	8270(3550)	2	10-150	≤50	140	1700
Phorate	8270(3550)	2	10-150	≤50	100	330
2-Picoline	8270(3550)	2	10-150	≤50	33	330
Pronamide	8270(3550)	2	10-150	≤50	38	330
Pyrene (MS)	8270(3550)	2	10-133	≤42	63	330
	8270(3550)-Low Level	2	10-133	≤42	1.7	6.7
Pyridine	8270(3550)	2	10-150	≤50	30	330
Safrole	8270(3550)	2	10-150	≤50	50	330
Strychnine	8270(3550)	2	10-150	≤50	820	3300
Sulfotepp	8270(3550)	2	10-150	≤50	100	330
1,2,4,5-Tetrachlorobenzene	8270(3550)	2	10-150	≤50	43	330
2,3,4,5-Tetrachlorophenol	8270(3550)	2	10-150	≤50	61	330
2,3,4,6-Tetrachlorophenol	8270(3550)	2	36-121	≤31	25	330
Tetrachlorophenols (2,3,4,5 + 2,3,4,6)	8270(3550)	2	NA	NA	NA	330
Thionazin	8270(3550)	2	10-150	≤50	100	330
o-Toluidine	8270(3550)	2	10-150	≤50	31	330
1,2,4-Trichlorobenzene (MS)	8270(3550)	2	10-112	≤22	22	330
2,4,5-Trichlorophenol	8270(3550)	2	25-130	≤36	29	330
2,4,6-Trichlorophenol	8270(3550)	2	41-130	≤30	20	330
Trichlorophenols (2,4,5 + 2,4,6)	8270(3550)	2	NA	NA	NA	330
o,o,o-Triethylphosphorothioate	8270(3550)	2	10-150	≤50	51	330
1,3,5-Trinitrobenzene	8270(3550)	2	10-150	≤50	21	330
Surrogate – Nitrobenzene-d5	8270(3550)	2	20-120	NA	NA	NA
Surrogate – 2-Fluorobiphenyl	8270(3550)	2	30-120	NA	NA	NA
Surrogate – p-Terphenyl-d14	8270(3550)	2	30-131	NA	NA	NA
Surrogate – Phenol-d5	8270(3550)	2	19-114	NA	NA	NA
Surrogate – 2-Fluorophenol	8270(3550)	2	16-113	NA	NA	NA
Surrogate – 2,4,6- Tribromophenol	8270(3550)	2	23-129	NA	NA	NA
Surrogate –Ortho Terphenyl	8270(3550)-low level	2	30-130	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY (% Rec)	PRECISION (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Polychlorinated Dibenzo-p-dioxin 2,3,7,8-substituted Congeners						
2,3,7,8-TCDD	8280	2	69-145	≤40	0.0069	0.50
Polychlorinated Dibenzo-p-dioxin and Dibenzofuran classes						
tetra-CDD (MS)	8280	2	69-145	≤40	0.12	0.50
tetra-CDF (MS)	8280	2	59-142	≤40	0.040	0.50
penta-CDD (MS)	8280	2	41-203	≤40	0.089	0.50
Penta-CDF (MS)	8280	2	55-146	≤40	0.068	0.50
hexa-CDD (MS)	8280	2	45-174	≤53	0.13	0.50
hexa-CDF (MS)	8280	2	50-154	≤46	0.051	0.50
hepta-CDD (MS)	8280	2	20-170	≤50	0.13	1.0
hepta-CDF (MS)	8280	2	20-170	≤50	0.063	1.0
octa-CDD (MS)	8280	2	20-170	≤50	0.14	1.0
octa-CDF (MS)	8280	2	20-170	≤50	0.13	1.0
Internal Standards						
2,3,7,8-tetra-CDD-13C12	8280	2	25-150	NA	NA	NA
octa-CDD-13C12	8280	2	25-150	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Chlordimeform (Galecron)	625(3550)	1/2	10-150	≤50	580	670
5-Chlordimeform	625(3550)	1/2	10-150	≤50	300	330
Dithiocarbamates (as Ziram)[5]	630(3540)	63/2	50-126	≤52	190	1000
Benomyl (as Carbendazim)	631	55/2	49-139	≤50	7.8	30
Aminocarb	632(3550)	13/2	50-150	≤50	2.2	50
Barban	632(3550)	13/2	85-128	≤25	8.3	20
Bromacil	632(3550)	13/2	66-120	≤25	14	40
Carbaryl (MS)	632(3550)	13/2	50-150	≤50	14	50
Carbofuran	632(3550)	13/2	23-109	≤31	23	50
Chlorpropham	632(3550)	13/2	50-150	≤50	11	20
Diuron (MS)	632(3550)	13/2	50-150	≤50	3.3	5.0
Fenuron	632(3550)	13/2	50-150	≤50	4.1	10
Fluometuron	632(3550)	13/2	50-150	≤50	5.0	10
Linuron	632(3550)	13/2	64-130	≤28	3.8	5.0
Methiocarb	632(3550)	13/2	50-150	≤50	13	50
Methomyl	632(3550)	13/2	50-150	≤50	28	200
Monuron	632(3550)	13/2	65-109	≤25	2.7	5.0
Neburon	632(3550)	13/2	50-150	≤50	2.9	5.0
Oxamyl	632(3550)	13/2	50-150	≤50	12	50
Propham	632(3550)	13/2	50-150	≤50	10	50
Propoxur	632(3550)	13/2	63-116	≤33	69	200
Siduron	632(3550)	13/2	50-150	≤50	15	20
Swep	632(3550)	13/2	50-150	≤50	4.9	20
Oryzalin	638(3550)	21/2	60-140	≤40	4.5	25

[5]The compounds determined as ziram by Method 630 include amobam, farbam, mancozeb, maneb, metham, nabam, polyram, and zineb.

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Acenaphthene (MS)	8310(3550)	2	11-144	≤35	8.3	50
Acenaphthylene	8310(3550)	2	10-139	≤40	8.7	20
Anthracene	8310(3550)	2	10-126	≤40	0.34	4.0
Benzo(a)anthracene	8310(3550)	2	12-135	≤40	1.2	4.0
Benzo(b)fluoranthene	8310(3550)	2	10-150	≤40	0.27	4.0
Benzo(k)fluoranthene	8310(3550)	2	10-159	≤40	0.54	4.0
Benzo(g,h,i)perylene	8310(3550)	2	10-120	≤40	1.4	10
Benzo(a)pyrene	8310(3550)	2	10-128	≤40	1.0	4.0
Chrysene (MS)	8310(3550)	2	10-199	≤40	1.0	4.0
Dibenzo(a,h)anthracene	8310(3550)	2	10-110	≤40	3.7	10
Fluoranthene	8310(3550)	2	56-136	≤28	1.4	10
Fluorene (MS)	8310(3550)	2	10-142	≤40	0.82	10
Indeno(1,2,3-cd)pyrene	8310(3550)	2	10-116	≤40	1.1	10
1-Methylnaphthalene	8310(3550)	2	10-125	≤40	12	20
2-Methylnaphthalene	8310(3550)	2	10-125	≤40	5.1	20
Naphthalene (MS)	8310(3550)	2	31-159	≤34	6.0	20
Phenanthrene	8310(3550)	2	10-155	≤40	0.77	4.0
Pyrene (MS)	8310(3550)	2	49-156	≤28	2.1	10
Surrogate - 4-Terphenyl-d14	8310(3550)	2	28-151	NA	NA	NA

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Acetaldehyde	8315	2	30-156	≤36	19	250
Formaldehyde	8315	2	30-170	≤47	23	100
Acrylamide	8316	2	27-120	≤50	220	1000
Aldicarb (Temik) (MS)	8318	2	44-114	≤50	0.79	10
Aldicarb sulfone	8318	2	58-118	≤50	1.0	10
Aldicarb sulfoxide	8318	2	33-143	≤50	1.0	10
Carbofuran (Furadan) (MS)	8318	2	53-123	≤50	1.7	10
Carbaryl (Sevin)	8318	2	56-126	≤50	1.6	10
Dioxacarb	8318	2	55-125	≤50	1.9	10
3-Hydroxycarbofuran	8318	2	60-120	≤50	1.6	10
Methiocarb (Mesurol)	8318	2	52-122	≤50	2.0	10
Methomyl (Lannate)	8318	2	54-114	≤50	1.7	10
Oxamyl (MS)	8318	2	45-161	≤50	1.8	10
Promecarb	8318	2	44-120	≤50	1.7	10
Propoxur (Baygon)	8318	2	46-116	≤50	1.5	10

TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2-Amino-4,6-dinitrotoluene	8330	2	50-150	≤30	130	250
4-Amino-2,6-dinitrotoluene	8330	2	58-126	≤30	150	500
1,3-Dinitrobenzene (MS)	8330	2	50-132	≤30	39	250
2,4-Dinitrotoluene (MS)	8330	2	54-120	≤30	39	250
2,6-Dinitrotoluene	8330	2	59-122	≤30	87	500
Diphenylamine	8330	2	65-140	≤30	16	100
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	8330	2	54-166	≤30	99	500
Methyl-2,4,6-trinitro-phenylnitramine (Tetryl)	8330	2	41-165	≤30	220	500
Nitrobenzene	8330	2	52-152	≤30	130	250
Nitroglycerin	8332	2	48-152	≤50	38	1000
n-Nitrosodiphenylamine	8330	2	55-121	≤30	32	100
2-Nitrotoluene (MS)	8330	2	52-138	≤40	77	250
3-Nitrotoluene	8330	2	69-137	≤30	140	250
4-Nitrotoluene	8330	2	54-166	≤30	130	250
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	8330	2	29-156	≤600	130	500
Pentaerythritol tetranitrate (PETN)	8330	2	50-150	≤30	34	1000
1,3,5-Trinitrobenzene	8330	2	47-123	≤30	50	250
2,4,6-Trinitrotoluene	8330	2	74-119	≤30	74	250
Surrogate - 3,4-Dinitrotoluene	8330	2	22-128	NA	NA	NA

TABLE 5.2 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Glyphosate	547/SL SOP	51	50-150	≤50	570	2500
Acrylic Acid	SL-SOP	68	25-150	≤50	1100	5000
Cyanuric acid	SL-SOP	102	50-150	≤50	150	2500
Ethylenethiourea	SL-SOP	104	40-120	≤40	7.6	50
Maleic Acid/Maleic Anhydride	SL-SOP	103	69-106	≤21	20	80
Nitrocellulose	SL-SOP	108	20-150	≤60	14000	50000
Phthalic acid/Phthalic anhydride	SL-SOP	103	30-142	≤40	5.6	100
Thiodiglycol	SL-SOP	112	40-128	≤50	400	1500

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Arsenic	1311/6010(3010)	2	75-125	≤20	0.0053	0.20
Barium	1311/6010(3010)	2	75-125	≤20	0.0012	1.0
Cadmium	1311/6010(3010)	2	75-125	≤20	0.00071	0. 10
Chromium	1311/6010(3010)	2	75-125	≤20	0.0017	0.20
Lead	1311/6010(3010)	2	75-125	≤20	0.0015	0.20
Mercury	1311/7470	2	80-120	≤20	0.0072	0.020
Selenium	1311/6010(3010)	2	75-125	≤20	0.0042	0.50
Silver	1311/6010(3010)	2	75-125	≤20	0.0019	0.10

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Endrin	1311/8081 (3510/3520)	2	41-158	≤25	0.00023	0.0050
Lindane	1311/8081 (3510/3520)	2	40-139	≤36	0.000052	0.0025
Methoxychlor	1311/8081 (3510/3520)	2	60-155	≤43	0.0016	0.025
Chlordane	1311/8081 (3510/3520)	2	54-140	≤30	0.0061	0.025
Toxaphene	1311/8081 (3510/3520)	2	12-130	≤30	0.062	0.25
Heptachlor	1311/8081 (3510/3520)	2	37-148	≤26	0.00011	0.0025
Heptachlor Epoxide	1311/8081 (3510/3520)	2	43-141	≤31	0.000088	0.0025
Surrogate - Tetrachloro-m-xylene (TCMX)	1311/8081 (3510/3520)	2	30-150	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	1311/8081 (3510/3520)	2	30-150	NA	NA	NA
Surrogate - Dibutyl chlorendate (DBC)	1311/8081 (3510/3520)	2	30-150	NA	NA	NA

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
2,4-D	1311/8151	2	11-154	≤78	0.0095	0.025
2,4,5-TP (Silvex)	1311/8151	2	10-100	≤66	0.00022	0.025
Surrogate - DCAA	1311/8151	2	27-133	NA	NA	NA

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Benzene	1311/8260 (5030)	2	62-135	≤16	0.0011	0.020
Carbon tetrachloride	1311/8260 (5030)	2	57-128	≤13	0.0021	0.020
Chlorobenzene	1311/8260 (5030)	2	72-127	≤22	0.0032	0.020
1,2-Dichloroethane	1311/8260 (5030)	2	65-131	≤23	0.0023	0.020
Chloroform	1311/8260 (5030)	2	62-130	≤20	0.0036	0.020
1,1-Dichloroethylene	1311/8260 (5030)	2	46-147	≤30	0.0018	0.020
Methylethyl ketone	1311/8260 (5030)	2	42-167	≤31	0.044	0.10
Trichloroethylene	1311/8260 (5030)	2	56-143	≤35	0.0011	0.020
Tetrachloroethylene	1311/8260 (5030)	2	60-148	≤24	0.0064	0.020
Vinyl chloride	1311/8260 (5030)	2	43-142	≤21	0.0048	0.040
Surrogate - Toluene-d8	1311/8260 (5030)	2	77-122	NA	NA	NA
Surrogate - p-Bromofluorobenzene	1311/8260 (5030)	2	74-126	NA	NA	NA
Surrogate - Dibromofluoromethane	1311/8260 (5030)	2	70-130	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	1311/8260 (5030)	2	70-130	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	1311/8260 (5030)	2	70-130	NA	NA	NA

NOTE: Table 5.3 lists the routine TCLP analytes. Leachates are routinely diluted prior to preparation and/or analysis since the TCLP regulatory threshold limi can be met using a diluted sample. If additional analytes are requested, the RL for liquid samples (Table 5.1) is reported with the appropriate dilution fact applied. The TCLP leaching fluid and extraction procedure may contribute background levels of some analytes when non-routine methods and analytes a performed on TCLP leachates. For example, TCLP Extraction Fluid #2 is prepared with a sodium acetate buffer that will contribute significant concentrations sodium. Calcium and boron have also been detected in the TCLP filters.

TABLE 5.3. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RL^ (mg/L)
Nitrobenzene	1311/8270 (3510/3520)	2	50-111	≤21	0.0016	0.050
Hexachlorobenzene	1311/8270 (3510/3520)	2	49-121	≤31	0.0095	0.050
1,4-Dichlorobenzene	1311/8270 (3510/3520)	2	27-103	≤31	0.0014	0.050
2,4-Dinitrotoluene	1311/8270 (3510/3520)	2	37-129	≤32	0.0020	0.050
Hexachlorobutadiene	1311/8270 (3510/3520)	2	27-97	≤30	0.0018	0.050
Hexachloroethane	1311/8270 (3510/3520)	2	26-86	≤35	0.0016	0.050
Pyridine	1311/8270 (3510/3520)	2	10-134	≤50	0.0046	0.25
2,4,5-Trichlorophenol	1311/8270 (3510/3520)	2	38-127	≤28	0.0037	0.050
2,4,6-Trichlorophenol	1311/8270 (3510/3520)	2	36-126	≤22	0.0018	0.050
Cresols	1311/8270 (3510/3520)	2	24-136	≤27	0.0036	0.050
Pentachlorophenol	1311/8270 (3510/3520)	2	19-148	≤33	0.020	0.25
Surrogate - Nitrobenzene-d5	1311/8270 (3510/3520)	2	34-130	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	1311/8270 (3510/3520)	2	36-124	NA	NA	NA
Surrogate - p-Terphenyl-d14	1311/8270 (3510/3520)	2	14-148	NA	NA	NA
Surrogate - Phenol-d5	1311/8270 (3510/3520)	2	25-128	NA	NA	NA
Surrogate - 2-Fluorophenol	1311/8270 (3510/3520)	2	29-121	NA	NA	NA
Surrogate - 2,4,6-Tribromophenol	1311/8270 (3510/3520)	2	29-143	NA	NA	NA

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RL^ (ug)
Aldrin	TO4/TO10	75	10-144	≤38	0.0041	0.050
alpha-BHC	TO4/TO10	75	22-101	≤40	0.0022	0.050
beta-BHC	TO4/TO10	75	12-120	≤40	0.0053	0.050
Gamma-BHC (Lindane)	TO4/TO10	75	12-138	≤37	0.0019	0.050
delta-BHC	TO4/TO10	75	10-142	≤47	0.0019	0.050
Technical Chlordane	TO4/TO10	75	45-119	≤40	0.061	0.50
alpha Chlordane	TO4/TO10	75	45-140	≤40	0.0028	0.050
Gamma Chlordane	TO4/TO10	75	11-141	≤40	0.0026	0.050
4,4'-DDD	TO4/TO10	75	28-134	≤50	0.0060	0.10
4,4'-DDE	TO4/TO10	75	34-121	≤23	0.0069	0.10
4,4'-DDT	TO4/TO10	75	29-134	≤26	0.0074	0.10
Dieldrin	TO4/TO10	75	28-137	≤30	0.0023	0.10
Endosulfan I	TO4/TO10	75	10-141	≤40	0.0027	0.050
Endosulfan II	TO4/TO10	75	10-141	≤65	0.0037	0.10
Endosulfan sulfate	TO4/TO10	75	26-144	≤50	0.0053	0.10
Endrin	TO4/TO10	75	33-149	≤32	0.0085	0.10
Endrin aldehyde	TO4/TO10	75	10-130	≤86	0.0079	0.10
Endrin ketone	TO4/TO10	75	29-112	≤31	0.011	0.10
Heptachlor	TO4/TO10	75	17-138	≤38	0.0025	0.050
Heptachlor epoxide	TO4/TO10	75	15-142	≤40	0.0085	0.050
Methoxychlor	TO4/TO10	75	24-152	≤40	0.032	0.50
Toxaphene	TO4/TO10	75	41-126	≤50	0.55	5.0
PCB-1016	TO4/TO10	75	34-137	≤44	0.15	1.0
PCB 1221	TO4/TO10	75	15-178	≤30	0.27	1.0
PCB 1232	TO4/TO10	75	10-215	≤30	0.21	1.0
PCB-1242	TO4/TO10	75	39-150	≤30	0.25	1.0
PCB-1248	TO4/TO10	75	38-158	≤30	0.18	1.0
PCB-1254	TO4/TO10	75	66-122	≤30	0.11	1.0
PCB-1260	TO4/TO10	75	58-150	≤30	0.12	1.0
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	TO4/TO10	75	10-114	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	TO4/TO10	75	27-128	NA	NA	NA

TABLE 5.4. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Acenaphthene	TO13	75	67-112	≤25	4.4	10
Acenaphthylene	TO13	75	60-102	≤25	4.1	10
Anthracene	TO13	75	70-130	≤25	3.8	10
Benzo(a)anthracene	TO13	75	73-101	≤25	2.7	10
Benzo(b)fluoranthene	TO13	75	70-130	≤25	3.0	10
Benzo(k)fluoranthene	TO13	75	70-130	≤25	4.9	10
Benzo(a)pyrene	TO13	75	74-104	≤25	2.9	10
Benzo(g,h,i)perylene	TO13	75	48-100	≤25	2.2	10
Benzyl alcohol	TO13	75	58-100	≤25	2.4	10
bis(2-Chloroethoxy)methane	TO13	75	66-100	≤25	2.5	10
bis(2-Chloroethyl)ether	TO13	75	50-100	≤25	2.2	10
bis(2-Chloroisopropyl)ether	TO13	75	62-100	≤25	3.3	10
bis(2-Ethylhexyl)phthalate	TO13	75	50-150	≤25	4.9	10
4-Bromophenyl phenyl ether	TO13	75	70-130	≤25	3.9	10
Butylbenzyl phthalate	TO13	75	50-150	≤25	2.8	10
4-Chloroaniline	TO13	75	62-100	≤25	2.6	20
4-Chloro-3-methyl phenol	TO13	75	70-130	≤25	2.6	10
2-chloronapthalene	TO13	75	69-100	≤25	2.8	10
2-Chlorophenol	TO13	75	53-100	≤25	2.7	10
4-Chlorophenyl phenyl ether	TO13	75	50-150	≤25	4.2	10
Chrysene	TO13	75	70-130	≤25	2.4	10
Dibenz(a,h)anthracene	TO13	75	55-100	≤25	2.4	10
Dibenzofuran	TO13	75	74-122	≤25	4.7	10
1,2-Dichlorobenzene	TO13	75	46-100	≤25	3.4	10
1,3-Dichlorobenzene	TO13	75	41-100	≤25	3.1	10
1,4-Dichlorobenzene	TO13	75	44-100	≤25	3.3	10
3,3'-Dichlorobenzidine	TO13	75	77-136	≤25	5.6	10
2,4-Dichlorophenol	TO13	75	69-100	≤25	2.7	10
Diethylphthalate	TO13	75	70-130	≤25	3.4	10

TABLE 5.4. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RL^ (ug)
2,4-Dimethylphenol	TO13	75	48-100	≤25	2.5	10
Dimethylphthalate	TO13	75	70-130	≤25	2.9	10
Di-n-butylphthalate	TO13	75	50-150	≤25	2.0	10
2,4-Dinitrotoluene	TO13	75	66-110	≤25	4.2	10
2,6-Dinitrotoluene	TO13	75	59-100	≤25	3.2	10
Di-n-octyl phthalate	TO13	75	50-150	≤25	5.9	10
Fluoranthene	TO13	75	70-130	≤25	3.8	10
Fluorene	TO13	75	70-130	≤25	5.1	10
Hexachlorobenzene	TO13	75	74-123	≤25	4.7	10
Hexachlorobutadiene	TO13	75	58-104	≤25	4.4	10
01Hexachloroethane	TO13	75	44-100	≤25	2.5	10
Indeno(1,2,3-cd)pyrene	TO13	75	59-100	≤25	2.6	10
Isophorone	TO13	75	52-100	≤25	3.9	10
2-Methyl naphthalene	TO13	75	24-107	≤25	8.0	10
2-Methylphenol	TO13	75	59-100	≤25	3.0	10
4-Methylphenol	TO13	75	59-109	≤25	4.8	10
Naphthalene	TO13	75	30-100	≤25	6.5	10
2-Nitroaniline	TO13	75	62-100	≤25	2.5	50
3-Nitroaniline	TO13	75	75-100	≤25	2.4	50
4-Nitroaniline	TO13	75	76-100	≤25	2.0	50
Nitrobenzene	TO13	75	20-100	≤25	5.0	10
2-Nitrophenol	TO13	75	35-100	≤25	2.4	10
N-nitrosodiphenylamine	TO13	75	70-130	≤25	2.8	10
N-nitrosodi-n-propylamine	TO13	75	63-100	≤25	3.0	10
Phenanthrene	TO13	75	70-130	≤25	3.6	10
Phenol	TO13	75	74-135	≤25	5.8	10
Pyrene	TO13	75	70-130	≤25	3.7	10
1,2,4-Trichlorobenzene	TO13	75	57-100	≤25	3.8	10
2,4,5-Trichlorophenol	TO13	75	77-121	≤25	4.2	10
2,4,6-Trichlorophenol	TO13	75	65-105	≤25	3.8	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RL▲ (ug)
Foramaldehyde	TO5	75	43-142	≤30	3.5	50
Formaldehyde	TO11	75	43-142	≤30	3.5	50

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (mg/m^3)	RL▲ (mg/m^3)
Dichlorodifluoromethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.77	2.0
Chloromethane	EPA 18 (Tedlar bag)	76	60-140	≤30	1.0	2.0
Vinyl chloride	EPA 18 (Tedlar bag)	76	60-140	≤30	0.51	2.0
Bromomethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.67	2.0
Chloroethane	EPA 18 (Tedlar bag)	76	60-140	≤30	1.0	2.0
Trichlorofluoromethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.51	2.0
1,1-Dichloroethene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.44	1.0
Methylene chloride (Dichloromethane)	EPA 18 (Tedlar bag)	76	60-140	≤30	0.26	1.0
Trans-1,2-Dichloroethene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.25	1.0
1,1-Dichloroethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.16	1.0
2,2-Dichloropropane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.25	1.0
Cis-1,2-Dichloroethene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.24	1.0
Chloroform	EPA 18 (Tedlar bag)	76	60-140	≤30	0.14	1.0
Bromochloromethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.25	1.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m^3)	RL^ (mg/m^3)
1,1,1-Trichloroethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.26	1.0
1,1-Dichloropropylene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.20	1.0
Carbon tetrachloride	EPA 18 (Tedlar bag)	76	60-140	≤30	0.27	1.0
1,2-Dichloroethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.28	1.0
Benzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.13	1.0
Trichloroethylene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.15	1.0
1,2-Dichloropropane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.11	1.0
Bromodichloromethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.13	1.0
Dibromomethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.14	1.0
Trans-1,3-Dichloropropene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.15	1.0
Toluene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.21	1.0
Cis-1,3-Dichloropropene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.10	1.0
1,1,2-Trichloroethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.15	1.0
1,3-Dichloropropane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.12	1.0
Tetrachloroethene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.28	1.0
Dibromochloromethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.21	1.0
1,2-Dibromoethane(EDB)	EPA 18 (Tedlar bag)	76	60-140	≤30	0.13	1.0
Chlorobenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.12	1.0
Ethylbenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.18	1.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m^3)	RL^ (mg/m^3)
m&p-Xylene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.44	1.0
o-Xylene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.27	1.0
Styrene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.20	1.0
Isopropylbenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.20	1.0
Bromoform	EPA 18 (Tedlar bag)	76	60-140	≤30	0.28	1.0
1,1,2,2-Tetrachloroethane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.24	1.0
1,2,3-Trichloropropane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.84	2.0
n-Propylbenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.19	1.0
Bromobenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.17	1.0
1,3,5-Trimethylbenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.22	1.0
2-Chlorotoluene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.36	1.0
4-Chlorotoluene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.22	1.0
t-Butylbenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.20	1.0
1,2,4-Trimethylbenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.27	1.0
s-Butylbenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.23	1.0
p-Isopropyltoluene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.23	1.0
1,3-Dichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.17	1.0
1,4-Dichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.17	1.0
n-Butylbenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.24	1.0
1,2-Dichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.16	1.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m^3)	RL^ (mg/m^3)
1,2-Dibromo-3-chloropropane	EPA 18 (Tedlar bag)	76	60-140	≤30	0.87	2.0
1,2,4-Trichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.21	1.0
Hexachlorobutadiene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.25	1.0
Naphthalene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.32	1.0
1,2,3-Trichlorobenzene	EPA 18 (Tedlar bag)	76	60-140	≤30	0.28	1.0
Acetone	EPA 18 (Tedlar bag)	76	60-140	≤30	0.52	10
2-Butanone (MEK)	EPA 18 (Tedlar bag)	76	60-140	≤30	1.5	10
Vinyl Acetate	EPA 18 (Tedlar bag)	76	60-140	≤30	0.59	2.0
4-Methyl-2-pentanone (MIBK)	EPA 18 (Tedlar bag)	76	60-140	≤30	0.64	10
2-Hexanone	EPA 18 (Tedlar bag)	76	60-140	≤30	0.94	10
Carbon disulfide	EPA 18 (Tedlar bag)	76	60-140	≤30	0.20	1.0
Methyl t-butyl ether (MTBE)	EPA 18 (Tedlar bag)	76	60-140	≤30	0.19	1.0
Total hydrocarbons	EPA 18 (Tedlar bag)	76	60-140	≤30	1.0	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/m^3)	RL^ (mg/m^3)
Nitrogen(TCD)	EPA 18 (Tedlar bag)	76	75-125	≤25	10000	10000
Oxygen(TCD)	EPA 18 (Tedlar bag)	76	75-125	≤25	10000	10000
Carbon dioxide(TCD)	EPA 18 (Tedlar bag)	76	75-125	≤25	10000	18000
Carbon monoxide(TCD)	EPA 18 (Tedlar bag)	76	75-125	≤25	100	570
Methane(TCD)	EPA 18 (Tedlar bag)	76	75-125	≤25	100	330
Methane (FID)	EPA 18 (Tedlar bag)	76	75-125	≤25	1.5	5.0
Ethane(TCD)	EPA 18 (Tedlar bag)	76	75-125	≤25	150	610
Ethane (FID)	EPA 18 (Tedlar bag)	76	75-125	≤25	1.5	5.0
Ethene (TCD)	EPA 18 (Tedlar bag)	76	75-125	≤25	150	670
Ethene (FID)	EPA 18 (Tedlar bag)	76	75-125	≤25	1.5	5.0
Propane (FID)	EPA 18 (Tedlar bag)	76	75-125	≤25	0.37	5.0
Butane(FID)	EPA 18 (Tedlar bag)	76	75-125	≤25	0.84	5.0
Pentane(FID)	EPA 18 (Tedlar bag)	76	75-125	≤25	0.95	5.0
Hexane(FID)	EPA 18 (Tedlar bag)	76	75-125	≤25	1.1	5.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug)	RLA (ug)
Hydrocarbons/Aaromatics						
Cyclohexane	NIOSH 1500/OSHA 07	98/99	75-125	≤25	2.5	10
Cyclohexene	NIOSH 1500/OSHA 07	98/99	75-125	≤25	2.5	10
n-Heptane	NIOSH 1500/OSHA 07	98/99	75-125	≤25	2.5	10
Methylcyclohexane	NIOSH 1500/OSHA 07	98/99	75-125	≤25	2.5	10
n-Octane	NIOSH 1500/OSHA 07	98/99	75-125	≤25	2.5	10
n-Pentane	NIOSH 1500/OSHA 07	98/99	75-125	≤25	2.5	10
Benzene	NIOSH 1500/1501 OSHA 07	98/99	75-125	≤25	2.5	10
Toluene	NIOSH 1500/1501 OSHA 07	98/99	75-125	≤25	2.5	10
Ethylbenzene	NIOSH 1501/OSHA 07	98/99	75-125	≤25	2.5	10
Xylene (total)	NIOSH 1501/OSHA 07	98/99	75-125	≤25	2.5	10
Styrene	NIOSH 1501/OSHA 09	98/99	75-125	≤25	2.5	10
Cumene	NIOSH 1501	98	75-125	≤25	2.5	10
Naphthalene	NIOSH 1501/OSHA 07	98/99	75-125	≤25	2.5	10
a-Methylstyrene	NIOSH 1501/OSHA 07	98/99	75-125	≤25	2.5	10
Vinyltoluene	NISOH 1501/OSHA 07	98/99	75-125	≤25	2.5	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY *(% Rec)	PRECISION *(% RPD)	MDL** (ug)	RLA (ug)
Halogenated Hydrocarbons						
Benzyl chloride	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
Bromoform	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
Carbon tetrachloride	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
Chlorobenzene	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
Chlorobromomethane	NIOSH 1003	98	75-125	≤25	2.5	10
Chloroform	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
1,2-Dichlorobenzene	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
1,4,-Dichlorobenzene	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
1,3-Dichlorobenzene	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
1,1,-Dichloroethane	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
1,2-Dichloroethane	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
cis-1,2-Dichloroethene	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
trans-1,2-Dichloroethene	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
Hexachloroethane	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
1,1,1-Trichloroethane	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
Tetrachloroethene	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
1,1,2-Trichloroethane	NIOSH 1003/OSHA 07	98/99	75-125	≤25	2.5	10
1,2,3-Trichloropropane	NIOSH 1003/OSHA 07	98	75-125	≤25	2.5	10
Methylene chloride	NISOH 1005	98	75-125	≤25	2.5	10
Vinyl chloride	NIOSH 1007	98	75-125	≤25	2.5	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Methanol (Methyl alcohol)	NIOSH 2000	98	75-125	≤25	2.5	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Biphenyl (Diphenyl) and Biphenyl oxide	NIOSH 2530	98	75-125	≤25	2.5	10
Acenapthene	NIOSH 5515	98	75-125	≤25	0.84	10
Acenapthylene	NIOSH 5515	98	75-125	≤25	0.83	10
Anthracene and Phenanthrene	NIOSH 5515	98	75-125	≤25	0.84	10
Benzo(a)anthracene and Chrysene	NIOSH 5515V	98	75-125	≤25	2.4	10
Benzo(a)pyrene	NIOSH 5515	98	75-125	≤25	0.88	10
Benzo (b) fluoranthene and Benzo(k) fluoranthene	NIOSH 5515	98	75-125	≤25	2.6	10
Benzo(g,h,i)perylene	NIOSH 5515	98	75-125	≤25	0.74	10
Fluoranthene	NIOSH 5515	98	75-125	≤25	0.85	10
Fluorene	NIOSH 5515	98	75-125	≤25	0.87	10
Indeno(1,2,3-cd) pyrene and Dibenz(a,h) anthracene	NIOSH 5515	98	75-125	≤25	0.79	10
Naphthalene	NIOSH 5515	98	75-125	≤25	0.78	10
Pyrene	NIOSH 5515	98	75-125	≤25	1.2	10

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND
METHOD DETECTION LIMITS (MDL) FOR AIR SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Aluminum (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	3.2	20
Antimony (ICP)	OSHA ID-125G	99	75-125	≤20	0.42	2.0
Antimony (GFAA)	OSHA ID 121	99	80-120	≤20	0.11	1.0
Arsenic (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.39	1.0
Beryllium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.11	0.50
Cadmium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.31	0.50
Cadmium (GFAA)	OSHA ID-121	99	80-120	≤20	0.0090	0.10
Calcium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	2.4	50
Chromium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.44	1.0
Chromium (GFAA)	OSHA ID-121	99	80-120	≤20	0.050	1.0
Cobalt (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.41	1.0
Copper (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.78	2.0
Iron (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	3.9	5.0
Lead (ICP)	NIOSH 7300/OSHA ID-125G	98/2	75-125	≤20	0.14	0.50
Lead (GFAA)	OSHA ID-121	99	80-120	≤20	0.14	0.50
Magnesium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	1.8	50
Manganese (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.18	1.0
Mercury (CVAA)	NIOSH 6009	98	80-120	≤20	0.0020	0.020
Molybdenum (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.23	1.0
Nickel (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	1.1	4.0

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/)	RLA (ug/)
Selenium(ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.46	1.0
Selenium (GFAA)	OSHA ID-121	99	80-120	≤20	0.13	1.0
Silver (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.20	1.0
Silver (GFAA)	OSHA ID-121	99	80-120	≤20	0.016	0.10
Sodium (ICP)	NIOSH 7300	98	75-125	≤20	8.3	50
Thallium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.46	1.0
Thallium (GFAA)	OSHA ID-121	99	80-120	≤20	0.14	1.0
Tin (ICP)	OSHA ID-125G	99	75-125	≤20	1.3	5.0
Titanium(ICP)	NIOSH 7300	98	70-130	≤20	0.25	1.0
Vanadium (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.23	1.0
Zinc (ICP)	NIOSH 7300/OSHA ID-125G	98/99	75-125	≤20	0.45	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.4 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR AIR

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug)	RLA (ug)
Chloride	NIOSH 7903	98	75-125	≤25	2.5	10
Fluoride	NIOSH 7903	98	75-125	≤25	0.25	1.0
Nitrate	NIOSH 7903	98	75-125	≤25	0.25	1.0
Sulfate	NIOSH 7903	98	75-125	≤25	0.13	50

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Aluminum (ICP)	6010(3050)	2	75-125	≤20	3.2	20
Antimony (ICP)	6010(3050)	2	75-125	≤20	0.42	5.0
Antimony (GFAA)	7041(3050)	2	80-120	≤20	0.11	1.0
Arsenic (ICP)	6010(3050)	2	75-125	≤20	0.39	1.0
Arsenic (GFAA)	7060(3050)	2	80-120	≤20	0.27	1.0
Barium (ICP)	6010(3050)	2	75-125	≤20	0.42	1.0
Beryllium (ICP)	6010(3050)	2	75-125	≤20	0.11	0.40
Boron(ICP)	6010(3050)	2	75-125	≤20	0.59	5.0
Cadmium (ICP)	6010(3050)	2	75-125	≤20	0.056	0.50
Cadmium (GFAA)	7131 (3050)	2	80-120	≤20	0.0090	0.10
Calcium (ICP)	6010(3050)	2	75-125	≤20	2.4	50
Chromium (ICP)	6010(3050)	2	75-125	≤20	0.18	1.0
Chromium (GFAA)	7191(3050)	2	80-120	≤20	0.050	1.0
Cobalt (ICP)	6010 (3050)	2	75-125	≤20	0.13	1.0
Copper (ICP)	6010 (3050)	2	75-125	≤20	0.22	2.0
Iron (ICP)	6010 (3050)	2	75-125	≤20	3.9	5.0
Lead (ICP)	6010 (3050)	2	75-125	≤20	0.20	0.50
Lead (GFAA)	7421(3050)	2	80-120	≤20	0.14	0.50
Magnesium (ICP)	6010 (3050)	2	75-125	≤20	1.8	50
Manganese (ICP)	6010(3050)	2	75-125	≤20	0.18	1.0
Mercury (CVAA)	7471	2	80-120	≤20	0.0020	0.020
Molybdenum (ICP)	6010(3050)	2	75-125	≤20	0.098	1.0
Nickel (ICP)	6010(3050)	2	75-125	≤20	1.1	4.0
Potassium (ICP)	6010(3050)	2	75-125	≤20	35	100
Selenium (ICP)	6010 (3050)	2	75-125	≤20	0.46	1.0
Selenium (GFAA)	7740(3050)	2	80-120	≤20	0.13	1.0

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Silver (ICP)	6010 (3050)	2	75-125	≤20	0.14	1.0
Silver (GFAA)	7761 (3050)	2	80-120	≤20	0.016	0.10
Sodium (ICP)	6010(3050)	2	75-125	≤20	8.3	50
Strontium(ICP)	6010(3050)	2	70-130	≤30	0.84	1.0
Thallium (ICP)	6010 (3050)	2	75-125	≤20	0.46	1.0
Thallium (GFAA)	7841 (3050)	2	80-120	≤20	0.14	1.0
Tin (ICP)	6010(3050)	2	75-125	≤20	1.3	5.0
Titanium(ICP)	6010(3050)	2	70-130	≤30	0.25	1.0
Vanadium (ICP)	6010 (3050)	2	75-125	≤20	0.23	1.0
Zinc (ICP)	6010 (3050)	2	75-125	≤20	0.45	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer
-T = trace ICP

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Cyanide, total	9012(9013)	2	75-125	≤30	0.59	1.0
	9012(9010)	2	75-125	≤30	0.14	1.0

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Aldrin (MS)	8081(3550)	97/2	10-144	≤38	0.021	1.7
Alpha-BHC	8081(3550)	97/2	22-101	≤40	0.088	1.7
Beta-BHC	8081(3550)	97/2	12-120	≤40	0.16	1.7
Gamma-BHC (Lindane) (MS)	8081(3550)	97/2	12-138	≤37	0.066	1.7
Delta-BHC	8081(3550)	97/2	10-142	≤47	0.18	1.7
Technical Chlordane	8081(3550)	97/2	45-119	≤40	1.7	17
Alpha Chlordane	8081(3550)	97/2	45-140	≤40	0.12	1.7
Gamma Chlordane	8081(3550)	97/2	11-141	≤40	0.078	1.7
4,4'-DDD	8081(3550)	97/2	28-134	≤50	0.22	3.3
4,4'-DDE	8081(3550)	97/2	34-121	≤23	0.13	3.3
4,4'-DDT (MS)	8081(3550)	97/2	29-134	≤26	0.49	3.3
Dieldrin (MS)	8081(3550)	97/2	28-137	≤30	0.19	3.3
Endosulfan I	8081(3550)	97/2	10-141	≤40	0.13	1.7
Endosulfan II	8081(3550)	97/2	10-159	≤65	0.22	3.3
Endosulfan sulfate	8081(3550)	97/2	26-144	≤50	0.91	3.3
Endrin (MS)	8081(3550)	97/2	33-149	≤32	0.10	3.3
Endrin aldehyde	8081(3550)	97/2	10-130	≤86	0.22	3.3
Endrin ketone	8081(3550)	97/2	29-112	≤31	0.42	3.3
Heptachlor (MS)	8081(3550)	97/2	17-138	≤38	0.12	1.7
Heptachlor epoxide	8081(3550)	97/2	15-142	≤40	0.099	1.7
Methoxychlor	8081(3550)	97/2	24-152	≤40	1.3	17
Toxaphene	8081(3550)	97/2	41-126	≤50	18	170
PCB-1016	8082(3550)	97/2	34-138	≤44	4.8	33
PCB 1221	8082(3550)	97/2	15-178	≤30	23	67
PCB 1232	8082(3550)	97/2	10-215	≤30	4.0	33
PCB-1242	8082(3550)	97/2	39-150	≤30	6.0	33
PCB-1248	8082(3550)	97/2	38-158	≤30	6.7	33
PCB-1254	8082(3550)	97/2	40-122	≤30	2.7	33
PCB-1260	8082(3550)	97/2	39-138	≤30	5.6	33
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081(3550)/8082(3550)	97/2	10-114	NA	NA	NA
Surrogate-Decachlorobiphenyl (DCB)	8081(3550)/8082(3550)	97/2	27-128	NA	NA	NA

SEVERN TRENT SERVICES

Biological Tissues Parameters

PARAMETER	METHOD	ACC (%REC)	PREC (%RPD)	MDL (ug/kg)	RL (ug/kg)
Semivolatiles by GC/MS					
Acenaphthene (MS)	8270(3550)	28-102	<=25	39	1000
Acenaphthylene	8270(3550)	54-140	<=25	39	1000
Anthracene	8270(3550)	39-106	<=30	36	1000
Benzo(a)anthracene	8270(3550)	29-111	<=25	29	1000
Benzoic acid	8270(3550)	10-150	<=50	63	5000
Benzo(b)fluoranthene	8270(3550)	19-126	<=25	51	1000
Benzo(k)fluoranthene	8270(3550)	26-125	<=38	39	1000
Benzo(g,h,i)perylene	8270(3550)	10-121	<=50	57	1000
Benzo(a)pyrene	8270(3550)	34-116	<=29	33	1000
Bis(2-chloroethoxy) methane	8270(3550)	13-108	<=50	90	1000
Bis(2-chloroethyl) ether	8270(3550)	10-130	<=50	29	1000
Bis(2-chloroisopropyl) ether	8270(3550)	36-166	<=50	30	1000
Bis(2-ethylhexyl) phthalate	8270(3550)	23-124	<=40	140	1000
4-Bromophenyl phenyl ether	8270(3550)	53-127	<=40	33	1000
Butyl benzyl phthalate	8270(3550)	24-120	<=40	90	1000
4-Chloroaniline	8270(3550)	10-150	<=50	51	2000
4-Chloro-3-methylphenol (MS) (p Chloro-m-cresol)	8270(3550)	25-107	<=25	30	1000
2-Chloronaphthalene	8270(3550)	60-118	<=40	33	1000
2-Chlorophenol (MS)	8270(3550)	23-114	<=25	33	1000
4-Chlorophenylphenyl ether	8270(3550)	20-118	<=33	30	1000
Chrysene	8270(3550)	35-130	<=27	36	1000
2-Methyl phenol (o-Cresol)	8270(3550)	14-95	<=50	39	1000
3- and 4-Methyl phenol (m and p-Cresol)	8270(3550)	12-102	<=50	180	1000
Dibenz(a,h)anthracene	8270(3550)	40-147	<=28	45	1000
Dibenzofuran	8270(3550)	10-150	<=50	36	1000
Di-n-butylphthalate	8270(3550)	28-125	<=50	45	1000
1,2-Dichlorobenzene	8270(3550)	10-99	<=40	33	1000
1,3-Dichlorobenzene	8270(3550)	10-100	<=42	26	1000
1,4-Dichlorobenzene (MS)	8270(3550)	10-125	<=40	33	1000
3,3'-Dichlorobenzidine	8270(3550)	10-189	<=100	48	2000
2,4-Dichlorophenol	8270(3550)	16-104	<=40	30	1000
Diethylphthalate	8270(3550)	31-113	<=40	45	1000
2,4-Dimethylphenol	8270(3550)	34973	<=25	30	1000
Dimethylphthalate	8270(3550)	20-116	<=40	33	1000
4,6-Dinitro-2-methylphenol	8270(3550)	43-135	<=93	87	5000
2,4-Dinitrophenol	8270(3550)	10-126	<=87	66	5000
2,4-Dinitrotoluene (MS)	8270(3550)	26-107	<=30	39	1000
2,6-Dinitrotoluene	8270(3550)	38-107	<=40	48	1000
Di-n-octylphthalate	8270(3550)	22-135	<=50	57	1000
Fluoranthene	8270(3550)	23-121	<=25	33	1000
Fluorene	8270(3550)	32-107	<=40	36	1000
Hexachlorobenzene	8270(3550)	44-105	<=40	30	1000
Hexachlorobutadiene	8270(3550)	10-112	<=40	33	1000
Hexachlorocyclopentadiene	8270(3550)	D-132	<=50	54	1000
Hexachloroethane	8270(3550)	16-91	<=40	39	1000

SEVERN TRENT SERVICES

Biological Tissues Parameters

SEVERN TRENT SERVICES

Biological Tissues Parameters

PARAMETER METHOD ACC (%REC) PREC (%RPD) MDL (ug/kg) RL (ug/kg)

PARAMETER	METHOD	ACC (%REC)	PREC (%RPD)	MDL (ug/kg)	RL (ug/kg)
Semivolatiles by GC/MS					
Indeno(1,2,3-cd)pyrene	8270(3550)	18-157	<=83	60	1000
Isophorone	8270(3550)	17-107	<=60	36	1000
2-Methylnaphthalene	8270(3550)	19-105	<=50	28	1000
Naphthalene	8270(3550)	53-125	<=25	33	1000
2-Nitroaniline	8270(3550)	22-110	<=50	42	5000
3-Nitroaniline	8270(3550)	10-117	<=50	33	5000
4-Nitroaniline	8270(3550)	10-136	<=50	39	5000
Nitrobenzene	8270(3550)	9-104	<=40	39	1000
2-Nitrophenol	8270(3550)	10-101	<=40	26	1000
4-Nitrophenol (MS)	8270(3550)	10-117	<=45	90	5000
N-Nitrosodiphenylamine	8270(3550)	40-122	<=50	290	1000
N-Nitroso-di-n-propylamine MS)	8270(3550)	11-117	<=35	42	1000
Pentachlorophenol (MS)	8270(3550)	10-120	<=44	84	5000
Phenanthrene	8270(3550)	38-113	<=25	36	1000
Phenol (MS)	8270(3550)	17-103	<=25	57	1000
Pyrene (MS)	8270(3550)	18-136	<=25	33	1000
1,2,4-Trichlorobenzene (MS)	8270(3550)	17-105	<=28	33	1000
2,4,5-Trichlorophenol	8270(3550)	39-123	<=27	39	1000
2,4,6-Trichlorophenol	8270(3550)	20-116	<=40	28	1000
Surrogates					
Nitrobenzene-d5	8270(3550)	12-125	NA	NA	NA
2-Fluorobiphenyl	8270(3550)	24-118	NA	NA	NA
p-Terphenyl-d14	8270(3550)	18-153	NA	NA	NA
Phenol-d5	8270(3550)	10-142	NA	NA	NA
2-Fluorophenol	8270(3550)	10-118	NA	NA	NA
2,4,6-Tribromophenol	8270(3550)	14-121	NA	NA	NA

RL based on an extraction weight of 10g and and a final extract volume of 1.0mL.

Biological Tissues Parameters

Biological Tissues Parameters

PARAMETER METHOD ACC (%REC) PREC (%RPD) MDL (ug/kg) RL (ug/kg)

PARAMETER	METHOD	ACC (%REC)	PREC (%RPD)	MDL (ug/kg)	RL (ug/kg)
Herbicides by GC/Electron Capture					
2,4-D (MS)	8151	19-153	<=47	4.8	25
Dalapon	8151	10-170	<=40	69	6000
2,4-DB	8151	20-160	<=40	19	25
Dicamba	8151	20-160	<=40	3.6	60
Dichlorprop	8151	30-170	<=40	8.1	300
Dinoseb	8151	10-130	<=50	25	300
MCPA	8151	10-130	<=50	2220	6000
MCPP	8151	10-130	<=50	1200	6000
2,4,5-T (MS)	8151	14-143	<=59	3	25
2,4,5-TP (Silvex) (MS)	8151	27-120	<=51	2.7	25
Surrogate					
2,4-Dichlorophenyl acetic acid (DCAA)	8151	30-189	NA	NA	NA

RL based on an extraction weight of 10g and and a final extract volume of 10mL.

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Monochlorobiphenyls	680/(3550)	97/93	30-130	≤50	0.68	10
Dihlorobiphenyls	680/(3550)	97/93	30-130	≤50	0.76	10
Trichlorobiphenyls	680/(3550)	97/93	30-130	≤50	0.68	10
Tetrachlorobiphenyls	680/(3550)	97/93	40-140	≤50	1.3	20
Pentachlorobiphenyls	680/(3550)	97/93	40-140	≤50	0.83	20
Hexachlorobiphenyls	680/(3550)	97/93	40-140	≤50	0.89	20
Heptachlorobiphenyls	680/(3550)	97/93	40-140	≤50	1.6	30
Octachlorobiphenyls	680/(3550)	97/93	40-140	≤50	0.95	30
Nonachlorobiphenyls	680/(3550)	97/93	30-130	≤50	1.9	50
Decachlorobiphenyl	680/(3550)	97/93	30-130	≤50	1.9	50
Surrogate- Decachlorobiphenyl-13C12	680/(3550)	97/93	30-130	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acetone	8260(5035)	97/2	14-189	≤40	2.3	50
	8260(5035ext)	97/2	14-189	≤40	230	5000
Benzene (MS)	8260(5035)	97/2	64-144	≤25	0.41	5.0
	8260(5035ext)	97/2	64-144	≤25	41	500
Bromodichloromethane	8260(5035)	97/2	71-140	≤40	0.34	5.0
	8260(5035ext)	97/2	71-140	≤40	34	500
Bromoform	8260(5035)	97/2	59-143	≤40	0.44	5.0
	8260(5035ext)	97/2	59-143	≤40	44	500
Bromomethane (Methyl bromide)	8260(5035)	97/2	35-181	≤65	2.0	10
	8260(5035ext)	97/2	35-181	≤65	200	1000
2-Butanone (MEK)	8260(5035)	97/2	54-166	≤40	2.8	25
	8260(5035ext)	97/2	54-166	≤40	280	2500
Carbon disulfide	8260(5035)	97/2	10-160	≤65	0.29	5.0
	8260(5035ext)	97/2	10-160	≤65	29	500
Carbon tetrachloride	8260(5035)	97/2	65-129	≤40	0.47	5.0
	8260(5035ext)	97/2	65-129	≤40	47	500
Chlorobenzene (MS)	8260(5035)	97/2	56-152	≤25	0.58	5.0
	8260(5035ext)	97/2	56-152	≤25	58	500
Chloroethane	8260(5035)	97/2	65-129	≤40	1.3	10
	8260(5035ext)	97/2	65-129	≤40	130	1000
Chloroform	8260(5035)	97/2	62-117	≤40	0.26	5.0
	8260(5035ext)	97/2	62-117	≤40	26	500
Chloromethane	8260(5035)	97/2	10-186	≤65	1.2	10
	8260(5035ext)	97/2	10-186	≤65	120	1000
Dibromochloromethane	8260(5035)	97/2	73-127	≤40	0.28	5.0
	8260(5035ext)	97/2	73-127	≤40	28	500
1,1-Dichloroethane	8260(5035)	97/2	64-112	≤40	0.28	5.0
	8260(5035ext)	97/2	64-112	≤40	28	500
1,2-Dichloroethane	8260(5035)	97/2	66-128	≤40	1.0	5.0
	8260(5035ext)	97/2	66-128	≤40	100	500
cis-1,2-Dichloroethene	8260(5035)	97/2	64-123	≤40	0.31	5.0
	8260(5035ext)	97/2	64-123	≤40	31	500
trans-1,2-Dichloroethene	8260(5035)	97/2	60-161	≤25	0.57	5.0
	8260(5035ext)	97/2	60-161	≤25	57	500
1,1-Dichloroethene (MS)	8260(5035)	97/2	44-157	≤25	0.77	5.0
	8260(5035ext)	97/2	44-157	≤25	77	500
1,2-Dichloropropane	8260(5035)	97/2	47-146	≤65	1.0	5.0
	8260(5035ext)	97/2	47-146	≤65	100	500

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
cis-1,3-Dichloropropene	8260(5035)	97/2	50-146	≤65	0.49	5.0
	8260(5035ext)	97/2	50-146	≤65	49	500
trans-1,3-Dichloropropene	8260(5035)	97/2	57-143	≤65	0.65	5.0
	8260(5035ext)	97/2	57-143	≤65	65	500
Ethylbenzene	8260(5035)	97/2	60-131	≤40	0.55	5.0
	8260(5035ext)	97/2	60-131	≤40	55	500
2-Hexanone	8260(5035)	97/2	47-145	≤40	3.7	25
	8260(5035ext)	97/2	47-145	≤40	370	2500
4-Methyl-2-pentanone (MIBK)	8260(5035)	97/2	54-167	≤49	3.2	25
	8260(5035ext)	97/2	54-167	≤49	320	2500
Styrene	8260(5035)	97/2	56-121	≤40	0.43	5.0
	8260(5035ext)	97/2	56-121	≤40	43	500
1,1,2,2-Tetrachloroethane	8260(5035)	97/2	36-157	≤40	0.80	5.0
	8260(5035ext)	97/2	36-157	≤40	80	500
Tetrachloroethene	8260(5035)	97/2	61-142	≤40	0.44	5.0
	8260(5035ext)	97/2	61-142	≤40	44	500
Toluene (MS)	8260(5035)	97/2	67-142	≤25	0.48	5.0
	8260(5035ext)	97/2	67-142	≤25	48	500
1,1,1-Trichloroethane	8260(5035)	97/2	11-148	≤40	0.98	5.0
	8260(5035ext)	97/2	11-148	≤40	98	500
1,1,2-Trichloroethane	8260(5035)	97/2	66-123	≤40	0.65	5.0
	8260(5035ext)	97/2	66-123	≤40	65	500

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Trichloroethene (MS)	8260(5035)	97/2	41-134	≤22	0.72	5.0
	8260(5035ext)	97/2	41-134	≤22	72	500
Vinyl chloride	8260(5035)	97/2	18-169	≤65	0.98	10
	8260(5035ext)	97/2	18-169	≤65	98	1000
Xylenes (total)	8260(5035)	97/2	50-150	≤40	1.5	5.0
	8260(5035ext)	97/2	50-150	≤40	150	500
o-Xylene	8260(5035)	97/2	22-154	≤40	0.50	5.0
	8260(5035ext)	97/2	22-154	≤40	50	500
m+p-Xylene	8260(5035)	97/2	62-123	≤40	1.0	5.0
	8260(5035ext)	97/2	62-123	≤40	100	500
Surrogate - Toluene-d8	8260(5035)	97/2	58-148	NA	NA	NA
	8260(5035ext)	97/2	58-148	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5035)	97/2	70-136	NA	NA	NA
	8260(5035ext)	97/2	70-136	NA	NA	NA
Surrogate -Dibromofluoromethane	8260(5035)	97/2	66-148	NA	NA	NA
	8260(5035ext)	97/2	66-148	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	8260(5035)	97/2	38-151	NA	NA	NA
	8260(5035ext)	97/2	38-151	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	8260(5035)	97/2	58-148	NA	NA	NA
	8260(5035ext)	97/2	58-148	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RL^ (ug/kg)
Acenaphthene (MS)	8270(3550)	97/2	28-102	≤25	13	330
Acenaphthylene	8270(3550)	97/2	54-140	≤25	13	330
Anthracene	8270(3550)	97/2	39-106	≤30	12	330
Benzo(a)anthracene	8270(3550)	97/2	29-111	≤25	9.6	330
Benzoic acid	8270(3550)	97/2	10-150	≤50	21	1700
Benzo(b)fluoranthene	8270(3550)	97/2	19-126	≤25	17	330
Benzo(k)fluoranthene	8270(3550)	97/2	26-125	≤38	13	330
Benzo(g,h,i)perylene	8270(3550)	97/2	10-121	≤50	19	330
Benzo(a)pyrene	8270(3550)	97/2	34-116	≤29	11	330
Bis(2-chloroethoxy) methane	8270(3550)	97/2	13-108	≤50	30	330
Bis(2-chloroethyl) ether	8270(3550)	97/2	10-130	≤50	9.6	330
Bis(2-chloroisopropyl) ether	8270(3550)	97/2	36-166	≤50	9.9	330
Bis(2-ethylhexyl) phthalate	8270(3550)	97/2	23-124	≤40	46	330
4-Bromophenyl phenyl ether	8270(3550)	97/2	53-127	≤40	11	330
Butyl benzyl phthalate	8270(3550)	97/2	24-120	≤40	30	330
4-Chloroaniline	8270(3550)	97/2	10-150	≤50	17	660
4-Chloro-3-methylphenol (MS) (p-Chloro-m-cresol)	8270(3550)	97/2	25-107	≤25	10	330
2-Chloronaphthalene	8270(3550)	97/2	60-118	≤40	11	330
2-Chlorophenol (MS)	8270(3550)	97/2	23-114	≤25	11	330
4-Chlorophenylphenyl ether	8270(3550)	97/2	20-118	≤33	10	330
Chrysene	8270(3550)	97/2	35-130	≤27	12	330
2-Methyl phenol (o-Cresol)	8270(3550)	97/2	14-95	≤50	13	330
3- Methyl phenol (m- Cresol)	8270(3550)	97/2	12-102	≤50	61	330
4-Methyl phenol (p-Cresol)	8270(3550)	97/2	12-102	≤50	61	330
3- and 4-Methyl phenol (m- and p-Cresol)	8270(3550)	97/2	12-102	≤50	61	330
Dibenz(a,h)anthracene	8270(3550)	97/2	40-147	≤28	15	330
Dibenzofuran	8270(3550)	97/2	10-150	≤50	12	330
Di-n-butylphthalate	8270(3550)	97/2	28-125	≤50	15	330
1,2-Dichlorobenzene	8270(3550)	97/2	10-99	≤40	11	330
1,3-Dichlorobenzene	8270(3550)	97/2	10-100	≤42	8.8	330
1,4-Dichlorobenzene (MS)	8270(3550)	97/2	10-125	≤40	11	330
3,3'-Dichlorobenzidine	8270(3550)	97/2	10-189	≤100	16	660
2,4-Dichlorophenol	8270(3550)	97/2	16-104	≤40	10	330
Diethylphthalate	8270(3550)	97/2	31-113	≤40	15	330
2,4-Dimethylphenol	8270(3550)	97/2	10-95	≤25	10	330
Dimethylphthalate	8270(3550)	97/2	20-116	≤40	11	330
4,6-Dinitro-2-methylphenol	8270(3550)	97/2	43-135	≤93	29	1700
2,4-Dinitrophenol	8270(3550)	97/2	10-126	≤87	22	1700

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,4-Dinitrotoluene (MS)	8270(3550)	97/2	26-107	≤30	13	330
2,6-Dinitrotoluene	8270(3550)	97/2	38-107	≤40	16	330
Di-n-octylphthalate	8270(3550)	97/2	22-135	≤50	19	330
Fluoranthene	8270(3550)	97/2	23-121	≤25	11	330
Fluorene	8270(3550)	97/2	32-107	≤40	12	330
Hexachlorobenzene	8270(3550)	97/2	44-105	≤40	10	330
Hexachlorobutadiene	8270(3550)	97/2	10-112	≤40	11	330
Hexachlorocyclopentadiene	8270(3550)	97/2	D-132	≤50	18	330
Hexachloroethane	8270(3550)	97/2	16-91	≤40	13	330
Indeno(1,2,3-cd)pyrene	8270(3550)	97/2	18-157	≤83	20	330
Isophorone	8270(3550)	97/2	17-107	≤60	12	330
2-Methylnaphthalene	8270(3550)	97/2	19-105	≤50	9.2	330
Naphthalene	8270(3550)	97/2	53-125	≤25	11	330
2-Nitroaniline	8270(3550)	97/2	22-110	≤50	14	1700
3-Nitroaniline	8270(3550)	97/2	10-117	≤50	11	1700
4-Nitroaniline	8270(3550)	97/2	10-136	≤50	13	1700
Nitrobenzene	8270(3550)	97/2	9-104	≤40	13	330
2-Nitrophenol	8270(3550)	97/2	10-101	≤40	8.8	330
4-Nitrophenol (MS)	8270(3550)	97/2	10-117	≤45	30	1700
N-Nitrosodiphenylamine/ Diphenylamine	8270(3550)	97/2	40-122	≤50	98	330
N-Nitroso-di-n-propylamine MS)	8270(3550)	97/2	11-117	≤35	14	330
Pentachlorophenol (MS)	8270(3550)	97/2	10-120	≤44	28	1700
Phenanthrene	8270(3550)	97/2	38-113	≤25	12	330
Phenol (MS)	8270(3550)	97/2	17-103	≤25	19	330
Pyrene (MS)	8270(3550)	97/2	18-136	≤25	11	330
1,2,4-Trichlorobenzene (MS)	8270(3550)	97/2	17-105	≤28	11	330
2,4,5-Trichlorophenol	8270(3550)	97/2	39-123	≤27	13	330
2,4,6-Trichlorophenol	8270(3550)	97/2	20-116	≤40	9.2	330
Surrogate - Nitrobenzene-d5	8270(3550)	97/2	12-125	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	8270(3550)	97/2	24-118	NA	NA	NA
Surrogate - p-Terphenyl-d14	8270(3550)	97/2	18-153	NA	NA	NA
Surrogate - Phenol-d5	8270(3550)	97/2	10-142	NA	NA	NA
Surrogate - 2-Fluorophenol	8270(3550)	97/2	10-118	NA	NA	NA
Surrogate - 2,4,6-Tribromophenol	8270(3550)	97/2	14-121	NA	NA	NA

TABLE 5.5 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD)	8280	97/2	69-145	≤40	0.13	0.50
Polychlorinated Dibenzo-p-dioxins and Dibenzofurans classes						
tetra-CDD	8280	97/2	69-145	≤40	0.12	0.50
tetra-CDF	8280	97/2	59-142	≤40	0.040	0.50
penta-CDD	8280	97/2	41-203	≤40	0.089	0.50
penta-CDF	8280	97/2	55-146	≤40	0.068	0.50
hexa-CDD	8280	97/2	45-174	≤53	0.13	0.50
hexa-CDF	8280	97/2	50-154	≤46	0.051	0.50
hepta-CDD	8280	97/2	20-170	≤50	0.13	1.0
hepta-CDF	8280	97/2	20-170	≤50	0.063	1.0
octa-CDD	8280	97/2	20-170	≤50	0.14	1.0
octa-CDF	8280	97/2	20-170	≤50	0.13	1.0
Internal Standard - 13C12-2,3,7,8-TCDD	8280	97/2	25-150	NA	NA	NA
Internal Standard - 13C12-2,3,7,8-TCDF	8280	97/2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,6,7,8-HxCDD	8280	97/2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,4,6,7,8-HpCDF	8280	97/2	25-150	NA	NA	NA
Internal Standard - 13C12-OCDD	8280	97/2	25-150	NA	NA	NA

TABLE 5.5. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR BIOLOGICAL TISSUES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acenaphthene (MS)	8310(3550)	97/2	11-144	≤35	14	50
Acenaphthylene	8310(3550)	97/2	10-139	≤40	5.1	20
Anthracene	8310(3550)	97/2	10-126	≤40	0.23	4.0
Benzo(a)anthracene	8310(3550)	97/2	12-135	≤40	0.27	4.0
Benzo(b)fluoranthene	8310(3550)	97/2	10-150	≤40	0.15	4.0
Benzo(k)fluoranthene	8310(3550)	97/2	10-159	≤40	0.12	4.0
Benzo(g,h,i)perylene	8310(3550)	97/2	10-120	≤40	0.68	10
Benzo(a)pyrene	8310(3550)	97/2	10-128	≤40	0.35	4.0
Chrysene (MS)	8310(3550)	97/2	10-199	≤40	0.28	4.0
Dibenzo(a,h)anthracene	8310(3550)	97/2	10-110	≤40	0.87	10
Fluoranthene	8310(3550)	97/2	56-136	≤28	0.47	10
Fluorene (MS)	8310(3550)	97/2	10-142	≤40	1.4	10
Indeno(1,2,3-cd)pyrene	8310(3550)	97/2	10-116	≤40	0.25	10
1-Methylnaphthalene	8310(3550)	97/2	10-125	≤40	6.2	20
2-Methylnaphthalene	8310(3550)	97/2	10-125	≤40	5.9	20
Naphthalene (MS)	8310(3550)	97/2	31-159	≤34	5.3	20
Phenanthrene	8310(3550)	97/2	10-155	≤40	0.50	4.0
Pyrene (MS)	8310(3550)	97/2	49-156	≤28	0.93	10
Surrogate - 4-Terphenyl-d14	8310(3550)	97/2	28-151	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Aluminum (ICP)	6010(3050)	98/2	75-125	≤20	3.2	20
Antimony (ICP)	6010(3050)	98/2	75-125	≤20	0.42	2.0
Antimony (GFAA)	7041(3050)	98/2	80-120	≤20	0.11	1.0
Arsenic (ICP)	6010(3050)	98/2	75-125	≤20	0.39	1.0
Arsenic (GFAA)	7060(3050)	98/2	80-120	≤20	0.27	1.0
Barium (ICP)	6010(3050)	98/2	75-125	≤20	0.42	1.0
Beryllium (ICP)	6010(3050)	98/2	75-125	≤20	0.11	0.40
Boron	6010(3050)	98/2	75-125	≤20	0.59	5.0
Cadmium (ICP)	6010(3050)	98/2	75-125	≤20	0.056	0.50
Cadmium (GFAA)	7131 (3050)	98/2	80-120	≤20	0.0090	0.10
Calcium (ICP)	6010(3050)	98/2	75-125	≤20	2.4	50
Chromium (ICP)	6010(3050)	98/2	75-125	≤20	0.18	1.0
Chromium (GFAA)	7191(3050)	98/2	80-120	≤20	0.050	1.0
Cobalt (ICP)	6010 (3050)	98/2	75-125	≤20	0.13	1.0
Copper (ICP)	6010 (3050)	98/2	75-125	≤20	0.22	2.0
Iron (ICP)	6010 (3050)	98/2	75-125	≤20	3.9	5.0
Lead (ICP)	6010 (3050)	98/2	75-125	≤20	0.20	0.50
Lead (GFAA)	7421(3050)	98/2	80-120	≤20	0.14	0.50
Magnesium (ICP)	6010 (3050)	98/2	75-125	≤20	1.8	50
Manganese (ICP)	6010(3050)	98/2	75-125	≤20	0.18	1.0
Mercury (CVAA)	7471	98/2	80-120	≤20	0.0020	0.020
Molybdenum (ICP)	6010(3050)	98/2	75-125	≤20	0.098	1.0
Nickel (ICP)	6010(3050)	98/2	75-125	≤20	1.1	4.0
Potassium (ICP)	6010(3050)	98/2	75-125	≤20	35	100
Selenium (ICP)	6010 (3050)	98/2	75-125	≤20	0.46	1.0
Selenium (GFAA)	7740(3050)	98/2	80-120	≤20	0.13	1.0

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Silver (ICP)	6010 (3050)	98/2	75-125	≤20	0.14	1.0
Silver (GFAA)	7761 (3050)	98/2	80-120	≤20	0.016	0.10
Sodium (ICP)	6010(3050)	98/2	75-125	≤20	8.3	50
Strontium(ICP)	6010(3050)	98/2	75-125	≤30	0.84	1.0
Thallium (ICP)	6010 (3050)	98/2	75-125	≤20	0.46	1.0
Thallium (GFAA)	7841 (3050)	98/2	80-120	≤20	0.14	1.0
Tin (ICP)	6010(3050)	98/2	75-125	≤20	1.3	5.0
Titanium(ICP)	6010(3050)	98/2	70-130	≤30	0.25	1.0
Vanadium (ICP)	6010 (3050)	98/2	75-125	≤20	0.23	1.0
Zinc (ICP)	6010 (3050)	98/2	75-125	≤20	0.45	2.0

ICP = inductively coupled (argon) plasma atomic emission spectrophotometer
GFAA = graphite furnace atomic adsorption spectrophotometer
CVAA = cold vapor atomic adsorption spectrophotometer

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL* (ug/wipe)	RLA (ug/wipe)
Aldrin (MS)	8081(3550)	98/2	10-144	≤38	0.0041	0.050
alpha-BHC	8081(3550)	98/2	22-101	≤40	0.0022	0.050
beta-BHC	8081(3550)	98/2	12-120	≤40	0.0053	0.050
gamma-BHC (Lindane) (MS)	8081(3550)	98/2	12-138	≤37	0.00119	0.050
delta-BHC	8081(3550)	98/2	10-142	≤47	0.0019	0.050
technical Chlordane	8081(3550)	98/2	45-119	≤40	0.061	0.20
alpha Chlordane	8081(3550)	98/2	45-140	≤40	0.0028	0.050
gamma Chlordane	8081(3550)	98/2	11-141	≤40	0.0026	0.050
4,4'-DDD	8081(3550)	98/2	28-134	≤50	0.0060	0.10
4,4'-DDE	8081(3550)	98/2	34-121	≤25	0.0069	0.10
4,4'-DDT (MS)	8081(3550)	98/2	29-134	≤26	0.0074	0.10
Dieldrin (MS)	8081(3550)	98/2	28-137	≤30	0.0023	0.10
Endosulfan I	8081(3550)	98/2	10-141	≤40	0.0027	0.050
Endosulfan II	8081(3550)	98/2	10-159	≤65	0.0037	0.10
Endosulfan sulfate	8081(3550)	98/2	26-144	≤50	0.0053	0.10
Endrin (MS)	8081(3550)	98/2	33-149	≤32	0.0085	0.10
Endrin aldehyde	8081(3550)	98/2	10-130	≤86	0.0079	0.10
Endrin ketone	8081(3550)	98/2	29-112	≤31	0.011	0.10
Heptachlor (MS)	8081(3550)	98/2	17-138	≤38	0.0025	0.050
Heptachlor epoxide	8081(3550)	98/2	15-142	≤40	0.0085	0.050
Methoxychlor	8081(3550)	98/2	24-152	≤40	0.032	0.50
Toxaphene	8081(3550)	98/2	41-126	≤50	0.55	5.0
PCB-1016	8082(3550)	98/2	34-138	≤44	0.15	1.0
PCB 1221	8082(3550)	98/2	15-178	≤30	0.27	2.0
PCB 1232	8082(3550)	98/2	10-215	≤30	0.21	1.0
PCB-1242	8082(3550)	98/2	39-150	≤30	0.25	1.0
PCB-1248	8082(3550)	98/2	38-158	≤30	0.18	1.0
PCB-1254	8082(3550)	98/2	40-122	≤30	0.11	1.0
PCB-1260	8082(3550)	98/2	39-138	≤30	0.12	1.0
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081(3550)/ 8082(3550)	98/2	10-114	NA	NA	NA
Surrogate-Decachlorobiphenyl (DCB)	8081(3550)/ 8082(3550)	98/2	27-128	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Monochlorobiphenyls	680/(3550)	98/93	30-130	≤50	0.044	0.30
Dihlorobiphenyls	680/(3550)	98/93	30-130	≤50	0.035	0.30
Trichlorobiphenyls	680/(3550)	98/93	30-130	≤50	0.035	0.30
Tetrachlorobiphenyls	680/(3550)	98/93	40--140	≤50	0.053	0.60
Pentachlorobiphenyls	680/(3550)	98/93	40--140	≤50	0.029	0.60
Hexachlorobiphenyls	680/(3550)	98/93	40--140	≤50	0.037	0.60
Heptachlorobiphenyls	680/(3550)	98/93	40--140	≤50	0.042	0.90
Octachlorobiphenyls	680/(3550)	98/93	40--140	≤50	0.064	0.90
Nonachlorobiphenyls	680/(3550)	98/93	30-130	≤50	0.11	1.5
Decachlorobiphenyl	680/(3550)	98/93	30-130	≤50	0.11	1.5
Surrogate- Decachlorobiphenyl-13C12	680/(3550)	98/93	30-130	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/wipe)	RL (ug/ wipe)
Acephate(1)	1657	98/72	25-140	≤50	1.2	5.0
Methamidophos(1)	1657	98/72	36-106	≤46	0.10	2.0
Alachlor	8141(3520)	98/2	46-139	≤30	0.14	1.0
Ametryn	8141(3520)	98/2	60-120	≤40	0.16	2.0
Atrazine (MS)	8141(3520)	98/2	39-130	≤30	0.13	2.0
Azinphos methyl	8141(3520)	98/2	48-162	≤50	0.16	1.0
Benoxacor	8141(3520)	98/2	10-150	≤50	4.4	10
Bolstar	8141(3520)	98/2	36-114	≤40	0.26	1.0
Butachlor	8141(3520)	98/2	50-150	≤40	0.35	1.0
Carbophenothion	8141(3520)	98/2	69-122	≤40	0.23	1.0
Chlordimeform (Galecron)	8141(3520)	98/2	10-150	≤50	3.8	10
5-Chloroaminotoluene	8141(3520)	98/2	10-150	≤50	3.4	10
Chlorpyrifos	8141(3520)	98/2	49-109	≤40	0.17	1.0
Chlorpyrifos methyl	8141(3520)	98/2	53-136	≤40	0.17	1.0
Coumaphos	8141(3520)	98/2	61-139	≤40	0.19	1.0
Demeton-o	8141(3520)	98/2	10-117	≤40	1.1	2.5
Demeton-s	8141(3520)	98/2	37-121	≤40	0.35	2.5
Demeton-o+s	8141(3520)	98/2	10-117	≤40	1.1	2.5
Diazinon (MS)	8141(3520)	98/2	40-137	≤40	0.17	1.0
Dichlofenthion	8141(3520)	98/2	38-118	≤40	0.11	1.0
Dichlorvos	8141(3520)	98/2	11-158	≤40	0.28	2.0
Dimethoate	8141(3520)	98/2	14-101	≤40	0.11	2.0
Dioxathion	8141(3520)	98/2	26-127	≤40	1.5	10
Disulfoton	8141(3520)	98/2	42-112	≤66	0.17	2.0
EPN	8141(3520)	98/2	48-124	≤40	0.19	1.0
Ethion	8141(3520)	98/2	62-175	≤40	0.12	0.50
Ethoprop	8141(3520)	98/2	42-123	≤40	0.27	0.50
Famphur	8141(3520)	98/2	13-128	≤60	0.25	2.0
Fenamiphos	8141	98/2	40-160	≤40	0.59	2.0
Fensulfothion	8141(3520)	98/2	31-163	≤40	0.27	5.0

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY * (% Rec)	PRECISION * (% RPD)	MDL** (ug/wipe)	RL^ (ug/ wipe)
Fenthion	8141(3520)	98/2	41-115	≤60	0.23	1.0
Isofenphos	8141	98/2	40-160	≤40	0.060	0.50
Malathion	8141(3520)	98/2	10-140	≤40	0.071	1.0
Merphos	8141(3520)	98/2	32-138	≤40	0.22	1.0
Metolachlor	8141(3520)	98/2	53-133	≤40	0.11	1.0
Metrabuzin	8141(3520)	98/2	50-150	≤40	0.11	1.0
Mevinphos	8141(3520)	98/2	24-166	≤40	0.23	2.0
Monocrotophos	8141(3520)	98/2	43-126	≤50	2.9	10
Naled	8141(3520)	98/2	10-119	≤40	0.14	5.0
Parathion, ethyl (MS)	8141(3520)	98/2	28-155	≤34	0.083	1.0
Parathion, methyl (MS)	8141(3520)	98/2	38-149	≤32	0.19	0.50
Phorate	8141(3520)	98/2	28-119	≤40	0.30	1.0
Prometon	8141(3520)	98/2	55-124	≤40	0.11	2.0
Prometryn	8141(3520)	98/2	36-155	≤40	0.23	2.0
Propazine	8141(3520)	98/2	51-127	≤30	0.25	2.0
Ronnel (MS)	8141(3520)	98/2	30-98	≤35	0.26	1.0
Simazine	8141(3520)	98/2	39-149	≤50	0.19	2.0
Stirophos (Tetrachlorvinphos)	8141(3520)	98/2	48-125	≤40	0.39	1.0
Sulfotepp	8141(3520)	98/2	40-157	≤40	0.17	0.50
Terbufos	8141(3520)	98/2	40-160	≤40	0.16	1.0
Terbutylazine	8141(3520)	98/2	60-130	≤40	0.33	2.0
Terbutryn	8141(3520)	98/2	53-113	≤40	0.066	2.0
Thionazin (MS)	8141(3520)	98/2	12-139	≤60	0.21	1.0
Tokuthion (Prothiofos)	8141(3520)	98/2	45-114	≤40	0.22	1.0
Trichloronate	8141(3520)	98/2	16-123	≤40	0.12	1.0
Surrogate - Triphenylphosphate	8141	98/2	16-164	NA	NA	NA

(1) Determined by NPD

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RLA (ug/wipe)
Acetone	8260(5030)	98/2	14-189	≤40	1.7	5.0
Benzene (MS)	8260(5030)	98/2	64-144	≤25	0.17	0.50
Bromodichloromethane	8260(5030)	98/2	71-140	≤40	0.17	0.50
Bromoform	8260(5030)	98/2	59-143	≤40	0.19	0.50
Bromomethane	8260(5030)	98/2	35-181	≤65	0.31	1.0
2-Butanone (MEK)	8260(5030)	98/2	54-166	≤40	2.6	2.5
Carbon disulfide	8260(5030)	98/2	10-160	≤65	0.090	0.50
Carbon tetrachloride	8260(5030)	98/2	65-129	≤40	0.25	0.50
Chlorobenzene (MS)	8260(5030)	98/2	56-152	≤25	0.18	0.50
Chloroethane	8260(5030)	98/2	65-129	≤40	0.65	1.0
Chloroform	8260(5030)	98/2	62-117	≤40	0.20	0.50
Chloromethane	8260(5030)	98/2	10-186	≤65	0.24	1.0
Dibromochloromethane	8260(5030)	98/2	73-127	≤40	0.17	0.50
1,1-Dichloroethane	8260(5030)	98/2	64-112	≤40	0.21	0.50
1,2-Dichloroethane	8260(5030)	98/2	66-128	≤40	0.20	0.50
cis-1,2-Dichloroethene	8260(5030)	98/2	64-123	≤40	0.25	0.50
trans-1,2-Dichloroethene	8260(5030)	98/2	60-161	≤25	0.32	0.50
1,1-Dichloroethene (MS)	8260(5030)	98/2	44-157	≤23	0.30	0.50
1,2-Dichloropropane	8260(5030)	98/2	47-146	≤65	0.21	0.50
cis-1,3-Dichloropropene	8260(5030)	98/2	50-146	≤65	0.18	0.50
trans-1,3-Dichloropropene	8260(5030)	98/2	57-143	≤65	0.18	0.50
Ethylbenzene	8260(5030)	98/2	60-131	≤40	0.18	0.50
2-Hexanone	8260(5030)	98/2	47-145	≤40	2.2	2.5
4-Methyl-2-pentanone (MIBK)	8260(5030)	98/2	54-167	≤49	1.8	2.5
Styrene	8260(5030)	98/2	56-121	≤40	0.20	0.50
1,1,2,2-Tetrachloroethane	8260(5030)	98/2	36-157	≤40	0.26	0.50
Tetrachloroethene	8260(5030)	98/2	61-142	≤40	0.23	0.50

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RL^ (ug/wipe)
Toluene (MS)	8260(5030)	98/2	67-142	≤23	0.16	0.50
1,1,1-Trichloroethane	8260(5030)	98/2	11-148	≤40	0.16	0.50
1,1,2-Trichloroethane	8260(5030)	98/2	66-123	≤40	0.20	0.50
Trichloroethene (MS)	8260(5030)	98/2	41-134	≤22	0.20	0.50
Vinyl chloride	8260(5030)	98/2	18-169	≤65	0.44	1.0
Xylenes (total)	8260(5030)	98/2	50-150	≤40	0.37	0.50
o-Xylene	8260(5030)	98/2	22-154	≤40	0.20	0.50
m+p-Xylene	8260(5030)	98/2	62-123	≤40	0.23	0.50
Surrogate - Toluene-d8	8260(5030)	98/2	58-148	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5030)	98/2	70-136	NA	NA	NA
Surrogate -Dibromofluoromethane	8260(5030)	98/2	66-148	NA	NA	NA
Surrogate - 1,2-Dichloroethane-d4	8260(5030)	98/2	38-151	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	8260(5030)	98/2	58-148	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL*** (ug/wipe)	RLA (ug/ wipe)
Acenaphthene (MS)	8270(3550)	98/2	28-102	≤25	0.71	10
Acenaphthylene	8270(3550)	98/2	54-140	≤25	1.8	10
Anthracene	8270(3550)	98/2	39-106	≤30	0.42	10
Benzo(a)anthracene	8270(3550)	98/2	29-111	≤25	0.59	10
Benzoic acid	8270(3550)	98/2	10-150	≤50	7.4	50
Benzo(b)fluoranthene	8270(3550)	98/2	19-126	≤25	0.64	10
Benzo(k)fluoranthene	8270(3550)	98/2	26-125	≤38	0.085	10
Benzo(g,h,i)perylene	8270(3550)	98/2	10-121	≤50	0.92	10
Benzo(a)pyrene	8270(3550)	98/2	34-116	≤29	0.58	10
Bis(2-chloroethoxy) methane	8270(3550)	98/2	13-108	≤50	1.7	10
Bis(2-chloroethyl) ether	8270(3550)	98/2	10-130	≤50	1.3	10
Bis(2-chloroisopropyl) ether	8270(3550)	98/2	36-166	≤50	1.3	10
Bis(2-ethylhexyl) phthalate	8270(3550)	98/2	23-124	≤40	0.61	10
4-Bromophenyl phenyl ether	8270(3550)	98/2	53-127	≤40	2.0	10
Butyl benzyl phthalate	8270(3550)	98/2	24-120	≤40	2.2	10
4-Chloroaniline	8270(3550)	98/2	10-150	≤50	1.4	20
4-Chloro-3-methylphenol (MS)	8270(3550)	98/2	25-107	≤25	0.82	10
2-Chloronaphthalene	8270(3550)	98/2	60-118	≤40	1.8	10
2-Chlorophenol (MS)	8270(3550)	98/2	23-114	≤25	1.4	10
4-Chlorophenylphenyl ether	8270(3550)	98/2	20-118	≤33	2.1	10
Chrysene	8270(3550)	98/2	35-130	≤27	0.46	10
2-Methyl phenol (o-Cresol)	8270(3550)	98/2	14-95	≤50	1.5	10
3- Methyl phenol (m- Cresol)	8270(3550)	98/2	12-102	≤50	1.5	10
4-Methyl phenol (p-Cresol)	8270(3550)	98/2	12-102	≤50	1.5	10
3- and 4-Methyl phenol (m- and p-Cresol)	8270(3550)	98/2	12-102	≤50	1.5	10
Dibenz(a,h)anthracene	8270(3550)	98/2	40-147	≤28	0.83	10
Dibenzofuran	8270(3550)	98/2	10-150	≤50	2.3	10
Di-n-butylphthalate	8270(3550)	98/2	28-125	≤50	0.65	10
1,2-Dichlorobenzene	8270(3550)	98/2	10-99	≤40	1.5	10
1,3-Dichlorobenzene	8270(3550)	98/2	10-100	≤42	1.3	10
1,4-Dichlorobenzene (MS)	8270(3550)	98/2	10-125	≤40	2.3	10
3,3'-Dichlorobenzidine	8270(3550)	98/2	10-189	≤100	8.0	20
2,4-Dichlorophenol	8270(3550)	98/2	16-104	≤40	1.6	10
Diethylphthalate	8270(3550)	98/2	31-113	≤40	0.86	10
2,4-Dimethylphenol	8270(3550)	98/2	10-95	≤25	2.1	10
Dimethylphthalate	8270(3550)	98/2	20-116	≤40	2.1	10
4,6-Dinitro-2-methylphenol	8270(3550)	98/2	43-135	≤93	3.9	50
2,4-Dinitrophenol	8270(3550)	98/2	10-126	≤87	2.8	50

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RL^ (ug/ wipe)
2,4-Dinitrotoluene (MS)	8270(3550)	98/2	26-107	≤30	1.5	10
2,6-Dinitrotoluene	8270(3550)	98/2	38-107	≤40	2.1	10
Di-n-octylphthalate	8270(3550)	98/2	22-135	≤50	0.63	10
Fluoranthene	8270(3550)	98/2	23-121	≤25	0.51	10
Fluorene	8270(3550)	98/2	32-107	≤40	0.60	10
Hexachlorobenzene	8270(3550)	98/2	44-105	≤40	0.44	10
Hexachlorobutadiene	8270(3550)	98/2	10-112	≤40	1.3	10
Hexachlorocyclopentadiene	8270(3550)	98/2	D-132	≤50	4.7	10
Hexachloroethane	8270(3550)	98/2	16-91	≤40	1.7	10
Indeno(1,2,3-cd)pyrene	8270(3550)	98/2	18-157	≤83	0.77	10
Isophorone	8270(3550)	98/2	17-107	≤60	1.9	10
2-Methylnaphthalene	8270(3550)	98/2	19-105	≤50	1.7	10
Naphthalene	8270(3550)	98/2	53-125	≤25	0.47	10
Naphthalene	8270(3550)	98/2	53-125	≤25	0.47	10
2-Nitroaniline	8270(3550)	98/2	22-110	≤50	2.2	50
3-Nitroaniline	8270(3550)	98/2	10-117	≤50	1.9	50
4-Nitroaniline	8270(3550)	98/2	10-136	≤50	2.0	50
Nitrobenzene	8270(3550)	98/2	9-104	≤40	1.6	10
2-Nitrophenol	8270(3550)	98/2	10-101	≤40	2.5	10
4-Nitrophenol (MS)	8270(3550)	98/2	10-117	≤45	2.4	50
N-Nitrosodiphenylamine/ Diphenylamine	8270(3550)	98/2	40-122	≤50	2.6	10
N-Nitroso-di-n-propylamine MS)	8270(3550)	98/2	11-117	≤35	1.4	10
Pentachlorophenol (MS)	8270(3550)	98/2	10-120	≤44	3.2	10
Phenanthrene	8270(3550)	98/2	38-113	≤25	0.92	10
Phenol (MS)	8270(3550)	98/2	17-103	≤25	1.0	10
Pyrene (MS)	8270(3550)	98/2	18-136	≤25	0.89	10
1,2,4-Trichlorobenzene (MS)	8270(3550)	98/2	17-105	≤28	1.4	10
2,4,5-Trichlorophenol	8270(3550)	98/2	39-123	≤27	1.9	10
2,4,6-Trichlorophenol	8270(3550)	98/2	20-116	≤40	1.8	10
Surrogate - Nitrobenzene-d5	8270(3550)	98/2	12-125	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	8270(3550)	98/2	24-118	NA	NA	NA
Surrogate - p-Terphenyl-d14	8270(3550)	98/2	18-153	NA	NA	NA
Surrogate - Phenol-d5	8270(3550)	98/2	10-142	NA	NA	NA
Surrogate - 2-Fluorophenol	8270(3550)	98/2	10-118	NA	NA	NA
Surrogate - 2,4,6-Tribromophenol	8270(3550)	98/2	14-121	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RL^ (ug/ wipe)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD)	8280	98/2	69-145	≤40	0.0012	0.0050
Polychlorinated Dibenzo-p-dioxins and Dibenzofurans classes						
tetra-CDD	8280	98/2	69-145	≤40	0.0012	0.0050
tetra-CDF	8280	98/2	59-142	≤40	0.0013	0.0050
penta-CDD	8280	98/2	41-203	≤40	0.0017	0.0050
penta-CDF	8280	98/2	55-146	≤40	0.0016	0.0050
hexa-CDD	8280	98/2	45-174	≤53	0.0011	0.0050
hexa-CDF	8280	98/2	50-154	≤46	0.0015	0.0050
hepta-CDD	8280	98/2	20-170	≤50	0.0018	0.0010
hepta-CDF	8280	98/2	20-170	≤50	0.0013	0.010
octa-CDD	8280	98/2	20-170	≤50	0.0013	0.010
octa-CDF	8280	98/2	20-170	≤50	0.0012	0.010
Internal Standard - 13C12-2,3,7,8-TCDD	8280	98/2	25-150	NA	NA	NA
Internal Standard - 13C12-2,3,7,8-TCDF	8280	98/2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,6,7,8-HxCDD	8280	98/2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,4,6,7,8-HpCDF	8280	98/2	25-150	NA	NA	NA
Internal Standard - 13C12-OCDD	8280	98/2	25-150	NA	NA	NA

TABLE 5.6 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WIPE SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/wipe)	RL^ (ug/wipe)
Acenaphthene (MS)	8310(3550)	98/2	11-144	≤35	0.38	1.0
Acenaphthylene	8310(3550)	98/2	10-139	≤40	0.17	1.0
Anthracene	8310(3550)	98/2	10-126	≤40	0.0060	0.20
Benzo(a)anthracene	8310(3550)	98/2	12-135	≤40	0.013	0.20
Benzo(b)fluoranthene	8310(3550)	98/2	10-150	≤40	0.0046	0.20
Benzo(k)fluoranthene	8310(3550)	98/2	10-159	≤40	0.0056	0.20
Benzo(g,h,i)perylene	8310(3550)	98/2	10-120	≤40	0.0035	0.50
Benzo(a)pyrene	8310(3550)	98/2	10-128	≤40	0.062	0.20
Chrysene (MS)	8310(3550)	98/2	10-199	≤40	0.012	0.20
Dibenzo(a,h)anthracene	8310(3550)	98/2	10-110	≤40	0.043	0.50
Fluoranthene	8310(3550)	98/2	56-136	≤28	0.014	0.50
Fluorene (MS)	8310(3550)	98/2	10-142	≤40	0.080	0.50
Indeno(1,2,3-cd)pyrene	8310(3550)	98/2	10-116	≤40	0.019	0.50
1-Methylnaphthalene	8310(3550)	98/2	10-125	≤40	0.15	1.0
2-Methylnaphthalene	8310(3550)	98/2	10-125	≤40	0.16	1.0
Naphthalene (MS)	8310(3550)	98/2	31-159	≤34	0.15	1.0
Phenanthrene	8310(3550)	98/2	10-155	≤40	0.015	0.20
Pyrene (MS)	8310(3550)	98/2	49-156	≤28	0.035	0.50
Surrogate - 4-Terphenyl-d14	8310(3550)	98/2	28-151	NA	NA	NA

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RL^ (mg/kg)
Aluminum (ICP)	6010(3050/3051)	2	75-125	≤20	3.2	20
Antimony (ICP)	6010(3050/3051)	2	75-125	≤20	0.42	2.0
Arsenic (ICP)	6010(3050/3051)	2	75-125	≤20	0.39	1.0
Arsenic (GFAA)	7060(3050/3051)	2	80-120	≤20	0.27	1.0
Barium (ICP)	6010(3050/3051)	2	75-125	≤20	0.42	1.0
Beryllium (ICP)	6010(3050/3051)	2	75-125	≤20	0.11	0.40
Cadmium (ICP)	6010(3050/3051)	2	75-125	≤20	0.056	0.50
Cadmium (GFAA)	7131 (3050/3051)	2	80-120	≤20	0.0090	0.10
Calcium (ICP)	6010(3050/3051)	2	75-125	≤20	2.4	50
Chromium (ICP)	6010(3050/3051)	2	75-125	≤20	0.44	1.0
Cobalt (ICP)	6010 (3050/3051)	2	75-125	≤20	0.13	1.0
Copper (ICP)	6010 (3050/3051)	2	75-125	≤20	0.22	2.0
Iron (ICP)	6010 (3050/3051)	2	75-125	≤20	0.88	5.0
Lead (ICP)	6010 (3050/3051)	2	75-125	≤20	0.20	0.50
Lead (GFAA)	7421(3050/3051)	2	80-120	≤20	0.14	0.50
Magnesium (ICP)	6010 (3050/3051)	2	75-125	≤20	1.7	50
Manganese (ICP)	6010(3050/3051)	2	75-125	≤20	0.18	1.0
Mercury (CVAA)	7471 (3050/3051)	2	80-120	≤20	0.0020	0.020
Molybdenum (ICP)	6010(3050/3051)	2	75-125	≤20	0.23	1.0
Nickel (ICP)	6010(3050/3051)	2	75-125	≤20	1.1	4.0
Potassium (ICP)	6010(3050/3051)	2	75-125	≤20	35	100
Selenium (ICP)	6010 (3050/3051)	2	75-125	≤20	0.46	1.0
Selenium (GFAA)	7740(3050/3051)	2	80-120	≤20	0.13	1.0
Silver (ICP)	6010 (3050/3051)	2	75-125	≤20	0.14	1.0
Silver (GFAA)	7761 (3050/3051)	2	80-120	≤20	0.016	0.10
Sodium (ICP)	6010(3050/3051)	2	75-125	≤20	8.3	50
Strontium(ICP)	6010(3050/3051)	2	75-125	≤30	0.84	1.0
Thallium (ICP)	6010 (3050/3051)	2	75-125	≤20	0.46	1.0
Thallium (GFAA)	7841 (3050/3051)	2	80-120	≤20	0.14	1.0
Tin (ICP)	6010(3050/3051)	2	75-125	≤20	1.3	5.0
Titanium	6010(3050/3051)	2	75-125	≤20	0.25	1.0
Vanadium (ICP)	6010(3050/3051)	2	75-125	≤20	0.23	1.0
Zinc (ICP)	6010(3050/3051)	2	75-125	≤20	0.45	2.0

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTE AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/kg)	RLA (mg/kg)
Cyanide, reactive	7.3.3.2	2	NA	≤50	NA	100mg HCN/ Kg Waste
Cyanide, total	9012(9013)	2	75-125	≤30	0.036	1.0
	9012(9010)	2	75-125	≤30	0.065	1.0
pH	9041	2	63-158	≤40	NA	NA

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Aldrin (MS)	8081(3580)	2	10-168	≤38	0.63	50
alpha-BHC	8081(3580)	2	37-134	≤40	2.6	50
beta-BHC	8081(3580)	2	17-147	≤40	4.8	50
gamma-BHC (Lindane) (MS)	8081(3580)	2	10-173	≤37	2.0	50
delta-BHC	8081(3580)	2	19-140	≤47	5.4	50
technical Chlordane	8081(3580)	2	45-119	≤40	51	500
alpha Chlordane	8081(3580)	2	45-140	≤40	3.6	50
gamma Chlordane	8081(3580)	2	45-140	≤40	2.3	50
4,4'-DDD	8081(3580)	2	31-140	≤50	6.6	100
4,4'-DDE	8081(3580)	2	30-145	≤25	3.9	100
4,4'-DDT (MS)	8081(3580)	2	10-181	≤26	15	100
Dieldrin (MS)	8081(3580)	2	10-176	≤30	5.7	100
Endosulfan I	8081(3580)	2	45-153	≤40	3.9	50
Endosulfan II	8081(3580)	2	10-202	≤65	6.6	100
Endosulfan sulfate	8081(3580)	2	26-144	≤50	27	100
Endrin (MS)	8081(3580)	2	10-180	≤32	3.0	100
Endrin aldehyde	8081(3580)	2	10-150	≤86	6.6	100
Endrin ketone	8081(3580)	2	40-100	≤31	13	100
Heptachlor (MS)	8081(3580)	2	10-162	≤38	3.6	50
Heptachlor epoxide	8081(3580)	2	37-142	≤40	3.0	50
Methoxychlor	8081(3580)	2	34-166	≤40	39	500
Toxaphene	8081(3580)	2	41-126	≤50	540	5000

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
PCB-1016	8082(3580)	2	34-138	≤44	140	1000
	EPA-600/4-81-045	61	34-138	≤44	1200	5000
PCB 1221	8082(3580)	2	15-178	≤30	690	1000
	EPA-600/4-81-045	61	15-178	≤30	1200	5000
PCB 1232	8082(3580)	2	10-215	≤30	120	1000
	EPA-600/4-81-045	61	10-215	≤50	1200	5000
PCB-1242	8082(3580)	2	39-150	≤30	180	1000
	EPA-600/4-81-045	61	39-150	≤30	1200	5000
PCB-1248	8082(3580)	2	38-158	≤30	200	1000
	EPA-600/4-81-045	61	38-158	≤30	1200	5000
PCB-1254	8082(3580)	2	40-122	≤30	81	1000
	EPA-600/4-81-045	61	40-122	≤30	1200	5000
PCB-1260	8082(3580)	2	39-138	≤30	170	1000
	EPA-600/4-81-045	61	39-138	≤30	1200	5000
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	8081(3580)/8082(3580)	2	10-114	NA	NA	NA
Surrogate - Decachlorobiphenyl (DCB)	8081(3580)/8082(3580)	2	27-128	NA	NA	NA

Waste dilution extraction : 1g of sample to a final volume of 10mL with appropriate solvent.

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Polychlorinated biphenyl classes						
Monochlorobiphenyls	680(3580)	93/2	30-130	≤50	20	300
Dichlorobiphenyls	680(3580)	93/2	30-130	≤50	23	300
Trichlorobiphenyls	680(3580)	93/2	30-130	≤50	20	300
Tetrachlorobiphenyls	680(3580)	93/2	40-140	≤50	39	600
Pentachlorobiphenyls	680(3580)	93/2	40-140	≤50	25	600
Hexachlorobiphenyls	680(3580)	93/2	40-140	≤50	27	600
Heptachlorobiphenyls	680(3580)	93/2	40-140	≤50	48	900
Octachlorobiphenyls	680(3580)	93/2	40-140	≤50	29	900
Nonochlorobiphenyls	680(3580)	93/2	30-130	≤50	57	1500
Decachlorobiphenyl	680(3580)	93/2	30-130	≤50	57	1500
Surrogate - $^{13}C_{12}$-Decachlorobiphenyl	680(3580)	93/2	30-130	NA	NA	NA

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acetone	8260(5035ext)	2	14-189	≤40	1200	25000
Benzene (MS)	8260(5035ext)	2	64-144	≤25	210	2500
Bromodichloromethane	8260(5035ext)	2	71-140	≤40	170	2500
Bromoform	8260(5035ext)	2	59-143	≤40	430	2500
Bromomethane (Methyl bromide)	8260(5035ext)	2	35-181	≤65	1000	5000
2-Butanone (MEK)	8260(5035ext)	2	54-166	≤40	1400	12500
Carbon disulfide	8260(5035ext)	2	10-160	≤65	150	2500
Carbon tetrachloride	8260(5035ext)	2	65-129	≤40	240	2500
Chlorobenzene (MS)	8260(5035ext)	2	56-152	≤25	290	2500
Chloroethane	8260(5035ext)	2	65-129	≤40	650	5000
Chloroform	8260(5035ext)	2	62-117	≤40	130	2500
Chloromethane	8260(5035ext)	2	10-186	≤65	600	5000
Dibromochloromethane	8260(5035ext)	2	73-127	≤40	140	2500
1,1-Dichloroethane	8260(5035ext)	2	64-112	≤40	140	2500
1,2-Dichloroethane	8260(5035ext)	2	66-128	≤40	500	2500
cis-1,2-Dichloroethene	8260(5035ext)	2	64-123	≤40	160	2500
trans-1,2-Dichloroethene	8260(5035ext)	2	60-161	≤25	290	2500
1,1-Dichloroethene (MS)	8260(5035ext)	2	44-157	≤25	390	2500
1,2-Dichloropropane	8260(5035ext)	2	47-146	≤65	500	2500
cis-1,3-Dichloropropene	8260(5035ext)	2	51-109	≤65	250	2500
trans-1,3-Dichloropropene	8260(5035ext)	2	57-143	≤65	330	2500
Ethylbenzene	8260(5035ext)	2	60-131	≤40	280	2500
2-Hexanone	8260(5035ext)	2	47-145	≤40	1900	12500
Methylene chloride	8260(5035ext)	2	61-149	≤65	330	2500
4-Methyl-2-pentanone (MIBK)	8260(5035ext)	2	64-167	≤49	1600	12500
Styrene	8260(5035ext)	2	56-121	≤40	2220	2500
1,1,2,2-Tetrachloroethane	8260(5035ext)	2	36-157	≤40	400	2500
Tetrachloroethene	8260(5035ext)	2	61-142	≤40	220	2500
Toluene (MS)	8260(5035ext)	2	67-142	≤25	240	2500
1,1,1-Trichloroethane	8260(5035ext)	2	11-148	≤40	490	2500
1,1,2-Trichloroethane	8260(5035ext)	2	66-123	≤40	330	2500
Trichloroethene (MS)	8260(5035ext)	2	41-134	≤25	360	2500
Vinyl chloride	8260(5035ext)	2	18-169	≤65	490	5000
Xylenes (total)	8260(5035ext)	2	50-150	≤40	750	2500
o-Xylene	8260(5035ext)	2	22-154	≤40	250	2500
m + p-Xylene	8260(5035ext)	2	62-123	≤40	500	2500
Surrogate - Toluene-d8	8260(5035ext)	2	58-148	NA	NA	NA
Surrogate - p-Bromofluorobenzene	8260(5035ext)	2	70-136	NA	NA	NA
Surrogate -Dibromofluoromethane	8260(5035ext)	2	66-148	NA	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	8260(5035ext)	2	70-130	NA	NA	NA

ext= 1g of waste to 10mL methanol; analyze 0.10mL of extract (equivalent to 0.010g of sample)

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
Acenaphthene (MS)	8270(3580)	2	28-102	≤25	3900	100000
Acenaphthylene	8270(3580)	2	54-140	≤25	3900	100000
Anthracene	8270(3580)	2	48-130	≤30	3600	100000
Benzo(a)anthracene	8270(3580)	2	42-143	≤25	2900	100000
Benzoic acid	8270(3580)	2	10-150	≤50	6300	500000
Benzo(b)fluoranthene	8270(3580)	2	49-123	≤25	5100	100000
Benzo(k)fluoranthene	8270(3580)	2	24-137	≤38	3900	100000
Benzo(g,h,i)perylene	8270(3580)	2	10-219	≤50	5700	100000
Benzo(a)pyrene	8270(3580)	2	44-141	≤29	3300	100000
Bis(2-chloroethoxy) methane	8270(3580)	2	33-184	≤50	9000	100000
Bis(2-chloroethyl) ether	8270(3580)	2	12-158	≤50	2900	100000
Bis(2-chloroisopropyl) ether	8270(3580)	2	36-166	≤50	3000	100000
Bis(2-ethylhexyl) phthalate	8270(3580)	2	10-158	≤40	14000	100000
4-Bromophenyl phenyl ether	8270(3580)	2	53-127	≤40	3300	100000
Butyl benzyl phthalate	8270(3580)	2	10-152	≤40	9000	100000
Carbazole	8270(3580)	2	10-150	≤50	4200	100000
4-Chloroaniline	8270(3580)	2	10-150	≤50	5100	200000
4-Chloro-3-methylphenol (MS) (p-Chloro-m-cresol)	8270(3580)	2	31-123	≤25	3000	100000
2-Chloronaphthalene	8270(3580)	2	60-118	≤40	3300	100000
2-Chlorophenol (MS)	8270(3580)	2	29-108	≤25	3300	100000
4-Chlorophenylphenyl ether	8270(3580)	2	25-158	≤33	3000	100000
Chrysene	8270(3580)	2	40-148	≤27	3600	100000
2-Methyl phenol (o-Cresol)	8270(3580)	2	10-150	≤50	3900	100000
3-Methyl phenol (m-Cresol)	8270(3580)	2	10-150	≤50	18000	100000
4-Methyl phenol (p-Cresol)	8270(3580)	2	10-150	≤50	18000	100000
Dibenz(a,h)anthracene	8270(3580)	2	40-147	≤28	4500	100000
Dibenzofuran	8270(3580)	2	10-150	≤50	3600	100000
Di-n-butylphthalate	8270(3580)	2	10-118	≤50	4500	100000
1,2-Dichlorobenzene	8270(3580)	2	32-129	≤40	3300	100000
1,3-Dichlorobenzene	8270(3580)	2	10-172	≤42	2600	100000
1,4-Dichlorobenzene (MS)	8270(3580)	2	20-114	≤40	3300	100000

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
3,3'-Dichlorobenzidine	8270(3580)	2	10-262	≤100	4800	200000
2,4-Dichlorophenol	8270(3580)	2	39-135	≤40	3000	100000
Diethylphthalate	8270(3580)	2	10-114	≤40	4500	100000
2,4-Dimethylphenol	8270(3580)	2	15-151	≤25	3000	100000
Dimethylphthalate	8270(3580)	2	10-112	≤40	3300	100000
4,6-Dinitro-2-methylphenol	8270(3580)	2	10-181	≤93	8700	500000
2,4-Dinitrophenol	8270(3580)	2	10-167	≤87	6600	500000
2,4-Dinitrotoluene (MS)	8270(3580)	2	25-128	≤30	3900	100000
2,6-Dinitrotoluene	8270(3580)	2	50-158	≤40	4800	100000
Diphenylamine/ N-nitrosodiphenylamine	8270(3580)	2	10-150	≤50	29000	100000
Fluoranthene	8270(3580)	2	54-135	≤21	3300	100000
Fluorene	8270(3580)	2	59-121	≤40	3600	100000
Hexachlorobenzene	8270(3580)	2	10-152	≤40	3000	100000
Hexachlorobutadiene	8270(3580)	2	24-116	≤40	3300	100000
Hexachlorocyclopentadiene	8270(3580)	2	10-150	≤50	5400	100000
Hexachloroethane	8270(3580)	2	40-113	≤40	3900	100000
Indeno(1,2,3-cd)pyrene	8270(3580)	2	18-157	≤83	6000	100000
Isophorone	8270(3580)	2	21-196	≤60	3600	100000
2-Methylnaphthalene	8270(3580)	2	10-150	≤50	2800	100000
Naphthalene	8270(3580)	2	53-125	≤25	3300	100000

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (prep)	REF	ACCURACY* (%Rec)	PRECISION * (%RPD)	MDL** (ug/kg)	RL^ (ug/kg
2-Nitroaniline	8270(3580)	2	10-150	≤50	4200	500000
3-Nitroaniline	8270(3580)	2	10-150	≤50	3300	500000
4-Nitroaniline	8270(3580)	2	10-150	≤50	3900	500000
Nitrobenzene	8270(3580)	2	35-180	≤40	3900	100000
2-Nitrophenol	8270(3580)	2	29-182	≤40	2400	100000
4-Nitrophenol (MS)	8270(3580)	2	10-125	≤45	9000	500000
N-Nitroso-di-n-propylamine (MS)	8270(3580)	2	27-140	≤35	4200	100000
Pentachlorophenol (MS)	8270(3580)	2	10-111	≤44	8400	500000
Phenanthrene	8270(3580)	2	56-129	≤25	3600	100000
Phenol (MS)	8270(3580)	2	26-108	≤25	5700	100000
Pyrene (MS)	8270(3580)	2	24-155	≤25	3300	100000
1,2,4-Trichlorobenzene (MS)	8270(3580)	2	23-124	≤28	3300	100000
2,4,5-Trichlorophenol	8270(3580)	2	39-123	≤27	3900	100000
2,4,6-Trichlorophenol	8270(3580)	2	37-144	≤40	2800	100000
Surrogate - Nitrobenzene-d5	8270(3580)	2	21-98	NA	NA	NA
Surrogate - 2-Fluorobiphenyl	8270(3580)	2	29-99	NA	NA	NA
Surrogate - p-Terphenyl-d14	8270(3580)	2	22-136	NA	NA	NA
Surrogate - Phenol-d5	8270(3580)	2	28-105	NA	NA	NA
Surrogate - 2-Fluorophenol	8270(3580)	2	18-107	NA	NA	NA
Surrogate - 2,4,6 - Tribromophenol	8270(3580)	2	12-122	NA	NA	NA

Waste dilution extraction : 1g of sample ot a final volume of 10mL with appropriate solvent.

TABLE 5.7 LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WASTES AND OILY SAMPLES

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (ug/kg)	RLA (ug/kg)
2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD)	8280	2	69-145	≤40	1.3	50
Polychlorinated Dibenzo-p-dioxins and Dibenzofurans classes						
tetra-CDD	8280	2	69-145	≤40	1.2	50
tetra-CDF	8280	2	59-142	≤40	0.40	50
penta-CDD	8280	2	41-203	≤40	0.89	50
penta-CDF	8280	2	55-146	≤40	0.68	50
hexa-CDD	8280	2	45-174	≤53	1.3	50
hexa-CDF	8280	2	50-154	≤46	0.51	50
hepta-CDD	8280	2	20-170	≤50	1.3	100
hepta-CDF	8280	2	20-170	≤50	0.63	100
octa-CDD	8280	2	20-170	≤50	1.4	100
octa-CDF	8280	2	20-170	≤50	1.3	100
Internal Standard - 13C12-2,3,7,8-TCDD	8280	2	25-150	NA	NA	NA
Internal Standard - 13C12-2,3,7,8-TCDF	8280	2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,6,7,8-HxCDD	8280	2	25-150	NA	NA	NA
Internal Standard - 13C12-1,2,3,4,6,7,8-HpCDF	8280	2	25-150	NA	NA	NA
Internal Standard - 13C12-OCDD	8280	2	25-150	NA	NA	NA

TABLE 5.8 FIELD ANALYTICAL METHODS, QA OBJECTIVES AND METHOD DETECTION LIMITS (MDL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REF	ACCURACY* (% Rec)	PRECISION* (% RPD)	MDL** (mg/L)	RLA (mg/L)
Chlorine, residual	4500-Cl F	4	NA	≤40	NA	0.05
	330.5/4500-Cl-G (Hach8021)	3/4	NA	≤40	NA	0.05
Hydrogen ion (pH)	150.1/9040	3/2	85-115	≤15	NA	NA
Oxygen (dissolved)	360.1	3	NA	≤30	NA	0.20
Salinity	2520B	4	NA	NA	NA	100
Specific conductance	120.1/9050	3/2	90-110	≤10	0.26 μS/cm	1.0 μS/cm
Temperature	170.1	3	NA	≤10	NA	NA
Turbidity	180.1/2130B	3/4	60-140	≤30	0.008 NTU	0.10 NTU
Water level	EPA	12	NA	≤5	NA	0.01 ft
Sulfite	4500-SO_3^{2-}B	4	75-125	≤30	NA	1.0
	377.1	3	75-125	≤30	NA	1.0

REFERENCES AND NOTES FOR TABLES IN SECTION 5

* Accuracy data are presented as recoveries for spikes or surrogates. For routine analysis of organics, percent recoveries are evaluated only on the subset spike compound lists specified by the methods. An (MS) following the parameter name designates the routine matrix and laboratory control spike compounds. Precision data are presented as relative percent difference (% RPD) and are advisory; i.e., not used for laboratory control. Since reportable levels (above detection limit) for most of the organic parameters may not be detected in all environmental samples, precision is usually based on duplicate spike data and evaluated according to method requirements.

Accuracy and precision control limits are primarily derived from in-house laboratory data. Some accuracy and precision limits have been rounded to the nearest "5". In some cases, method limits may be used in lieu of in-house limits because in-house limits are broader than the method limits or are too broad to be usable. These cases include all metals methods, certain of the 500-series methods, method 8015 DAI, and all 600 series methods. In-house accuracy and precision limits in Tables 5.1 - 5.8 have been developed from spike concentrations between 0.5 times to 10 times the routine reporting (quantitation) limits.

** Method Detection Limit (MDL)

^ Routine Reporting Limit (also referred to as quntitation limt or pratical quantitation limit(PQL).)

NA Not Applicable

SIM (Selected ion monitoring.) Savannah Laboratories has verified that the analytes listed can be determined using this procedure.

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89. EPA 600/R-95/131: *"Methods for the Determination of Organic Compounds in Drinking Water, Supplement III"*, August 1995.

90. National Council of the Paper Industry for Air and Stream Improvement: Special Report No. 95-07, Method 253, May 1995.

91. EPA 600/4-88-039: *"Methods for the Determination of Organic Compounds in Drinking Water,* Method 504, Revision 2.0(1989).

92. *Method 1650: "Absorbable Organic Halides by Adsorption in Microcoulometric Titrations; "Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Water and Wastewater"*: U.S. EPA Office of Water, Engineering, and Analysis Division, Washington, D.C..

93. *Method 680: "Determination of Pesticides and PCBs in Water and Soil/Sediment by GC/MS"*; U.S. EPA, Washington, D.C..

94. *Method 1653: "Chlorinated Phenolics in Water by In-situ Acetylation/GC/MS Determination"*, Method cp. 86.01, National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Avenue, NY 10016 (July 1986).

95. SL SOP AR30: *"Dissolved Gases in Water"*, based on *"Dissolved Oxygen and Methane in Water by a GC Headspace Equilibration Technique"* by Kampbell and Wilson; U.S. EPA. (March 1989).

96. SL SOP CU16: *"Wipe Test: Sampling"*, based on guidance from NIOSH and OSHA manuals.

97. *"Extraction and Analysis of Organics in Biological Tissues"*; U.S. EPA Environmental Services Division, Analytical Support Branch, Athens, GA (Method OB 10/90).

98. NIOSH Manual of Analytical Methods, Fourth Edition, August 1994, U.S. Department of Health and Human Services, Cincinnati, Ohio.

99. OSHA Analytical Methods Manual, Second Edition, January 1990. Department of Labor, Salt Lake City, Utah.

100. SL SOP ME 80: *Determination of Lithium by Flame Emission Spectroscopy.*

101. SL SOP VG41: *Direct Aqueous Injection (DAI) of Pulp and Paper Samples*
(This SOP is based on the guidance in NCASI draft Method *Methanol in Process Liquids by GC/FID.*

101. SL SOP LC67: *Determination of Cyanuric Acid in Water and Soil by HPLC*

102. SL SOP LC65:*Determination of Phthalic Acid and Maleic Acid in Water and Soils by HPLC*

104. SL SOP LC60: *Ethylenethiourea in Water and Soils*

105 SL SOP LC45: *Determination of Asulam in Water by HPLC*

106. *Method for the Determination of Extractable Petroleum Hydrocarbons (EPH)*; Massachusetts Department of Environmental Protection; January 1998

107. *Method for the Determination of Volatile Petroleum Hydrocarbons (VPH)*; Massachusetts Department of Environmental Protection; January 1998

108. SL SOP GE105: *Nitrocellulose in Water and Soil*

109. SL SOP LC42: *Determination of Nitroaromatics and Nitramines (Explosive Residues) in Water and Soil by HPLC (Modified 8330)*

110. ASTM Method D4282-83

111. ASTM Method 1385

112. SL SOP LC35: *Thiodiglycol by HPLC*

113. SL SOP GE114: *Perchlorate by Ion Chromatography*

114. TPH Method 1005, Texas Department of Natural Resources

6.0 SAMPLING PROCEDURES

When STL Savannah Laboratories is contracted to provide sampling services, a field crew is assigned to each project. Each crew is composed of experienced field sampling technicians and a highly qualified field sampling manager who is trained in EPA protocols for groundwater and other environmental sampling. On numerous past projects, these manager have had their field sampling techniques audited by Florida DEP, Georgia EPD, Alabama ADEM, South Carolina DHEC, and EPA Region IV QA or field personnel. The field sampling crews at Savannah Laboratories are responsible for collection, handling, field screening, documentation, and packaging and shipment of samples to the lab in accordance with client requests.

The crew adheres to the sampling protocol defined by the appropriate regulatory agency or the requirements defined in the project sampling plan (QAPP). In some cases, sampling procedures may be modified to comply with project requirements.

6.1 Sampling Capabilities

Savannah Laboratories has the capability for sampling groundwater, surface water, wastewater, soils, sediments/sludges, drinking water, and tissues for the following analyte classes:

Analyte Class	Sample Source
Volatile Organics (VOAs)	Drinking water, groundwater, surface water, wastewater, sols, sediments, fish, shellfish, plant and animal tissues, liquid hazardous wastes, sludges, solid and hazardous wastes, and domestic waste sludges.
Extractable Organics	Drinking water, groundwater, surface water, wastewater, soils, sediments, fish, shellfish, plant and animal tissues, liquid hazardous wastes, sludges, solid and hazardous wastes, and domestic waste sludges.
Metals	Drinking water, groundwater, surface water, wastewater, soils, sediments, fish, shellfish, plant and animal tissues, liquid hazardous wastes, sludges, solid and hazardous wastes, and domestic waste sludges.
Microbiology	Drinking water, groundwater, surface water, wastewater, soils, sediments, and tissues
Cyanide/Sulfide	Drinking water, groundwater, surface water, wastewater, soils, sediments, liquid hazardous wastes, sludges, solid and hazardous waste, and domestic waste sludges.
Inorganic Anions	Drinking water, groundwater, surface water, wastewater, soils, sediments, liquid hazardous wastes, sludges, solid and hazardous waste, and domestic waste sludges.
Organics: TOC, COD, BOD, Total Recoverable Petroleum Hydrocarbons, Oil & Grease, Phenolics, MBAS	Drinking water, groundwater, surface water, wastewater, soils, sediments, liquid hazardous wastes, sludges, solid and hazardous waste, and domestic waste sludges.
Physical Properties: Color, Specific Conductance, Hardness, Odor, pH, Residues, Temperature, Turbidity	Drinking water, groundwater, surface water, wastewater, soils, sediments, liquid hazardous wastes, sludges, solid and hazardous waste, and domestic waste sludges.

6.2 Sampling Equipment

The following is a list of equipment and the sample preservation reagents employed by Savannah Laboratories' field sampling crews.

Routinely Used Equipment	Use
Ice chests, Styrofoam or insulated plastic	Sample container and sample transport
Sampling vehicles	Sample container and sample transport
Field thermometer	Field measurement of temperature
Field pH meter	Field measurement of pH
Field conductivity meter	Field measurement of conductivity
Electronic water level indicator	Well volume calculation
Stainless steel tape measure	Well volume calculation
Nylon, monofilament, or polypropylene line	Well volume calculation
Sheet plastic	Contamination control
Aluminum foil	Contamination control
Plastic or metal buckets	Collection of purge water or cleaning wastes
Cleaning brushes	Equipment decontamination
Liquinox detergent in original container	Equipment decontamination
Analyte free water contained in contaminant-free glass or plastic bottles	Equipment decontamination
Isopropyl alcohol (nanograde) contained in contaminant-free glass bottles or Teflon squeeze bottles	Equipment decontamination
10% Nitric acid (metals grade) contained in contaminant-free glass bottles	Equipment decontamination (except for stainless steel equipment)
Glass or plastic bottles and dispensers	Equipment decontamination
Glass or plastic jugs	Transport of cleaning wastes
Field carrier (covered, divided tray or box)	Transport of preservation reagents
Narrow range pH paper	Field check of sample preservation
Disposable pipettes glass (organic) and plastic (inorganic)	Addition of preservation reagents
Standard buffer solutions (pH 4,7, and 10)	Calibration of field pH meter
Standard KCl solution(100, 500, 1000, 1413, 12880 micro ohms/cm)	Calibration check of field conductivity and salinity meter
Disposable unpowdered latex gloves	Contamination control
Ice	Sample preservation
Sealing Tape	Sealing sample containers (except VOA vials)
Shipping labels and forms	Shipping samples
Sample container labels	Labeling samples
Bubble pack	Packing samples
Clothing and goggles	Sampling safety
Notebooks	Documentation
Waterproof pens, markers	Documentation, labeling
Custody seals	Monitor for tampering
Custody forms	Document custody
Camera	Document site
Calculator	Calculations
Site maps	Determine locations
SOPs	Reference procedures
MSDS Sheet on all chemicals	Safety emergency
Turbidity meter with 0.75 , 10, and 100 NTU standards	Field measurement of turbidity
Chlorine test kit with pocket colorimeter	Field determination of chlorine
Sulfite test kit with calibration burets	Field determination of sulfite
Paper towels	General use

Preservation Reagents	Grade
HCl, 1:1	Metals grade, pre-assayed
HNO_3, 1:1	Metals grade, pre-assayed
H_2SO_4, 1:1	Metals grade, pre-assayed
NaOH	ACS reagent grade
$Na_2S_2O_3$, 10%, 0.008%	ACS reagent grade
Zn $(C_2H_3O_2)_2$, 2N	ACS reagent grade
Ascorbic acid, neat; 0.06%	ACS reagent grade
Chloroacetic acid	ACS reagent grade

6.3 Routine Sample Containers

The following tables list the parameter, the routine container, the chemical preservative required to maintain the integrity of the sample, the hold time (preparation and analytical), and the minimum volume or weight of sample required to complete the analysis.

The Routine Containers used for sampling and analysis are certified by sTL-SL to ensure that the parameters tested are below the reporting limits published in the current STL-SL LQM. The procedures for certifying containers are given in STL-SL SOP CU35: *Procedures for Contaminant-free Containers*. The preparation of sampling kits is described in STL-SL SOP CU15: *Preparation of Sampling Containers*.

The chemical preservative is the solution added to the sampling containers or supplied as separate solutions or neat materials that preserve the integrity of the sample. Chemical preservatives are generally analyte specific. Generally, parameters requiring the same type of container and preservation can be analyzed from the same container. In addition to chemical preservation, the samples must be iced at the time of collection and maintained in the laboratory at the required temperature, usually 0-4C (control limits of less than 6C with no frozen samples).

The hold time is the maximum time from collection that the sample can be held prior to preparation or analysis. Some parameters, for example, semivolatile organics, have separate hold times for preparation of the sample and analysis of the extract.

The routine reporting limit for some analyses can be attained when a less than optimum sample volume or weight is supplied. For some analyses (e.g., organic extractions), the final volume of the extract or digest can be adjusted to met the routine reporting limits. If a client requires a normal reporting limit from a reduced sample amount, the client must inform their project manager, who will alert the lab prior to sample analysis because the amount of surrogate and/or spiking solution must be adjusted prior to sample preparation.

The minimum volume or weight required for TCLP is determined by the procedure. The lab must have these minimum amounts of sample in order to meet the Regulatory Threshold Limits for hazardous wastes. If less than the required weight or volume is supplied, the data may be flagged or the results for the TCLP invalidated by the regulatory agency.

Table 1 - Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Water Matrices

General Chemistry

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Absorbable organic halides (AOX)	1650	500-mL Amb G	2mL 1:1 nitric acid	28 days
Acidity	305.1/305.2/SM 2310B	120-ml P	None	14 days
Alkalinity	310.1/SM2320B	120-ml P	None	14 days
Ammonia	(Clorimetric – 350.1/ SM4500-NH3-H) (Electrode – 350.2/ 350.3/SM4500-NH3-F)	120-ml P	0.5mL 1:1 sulfuric acid	28 days
Ammonia (if distillation required)	(Clorimetric – 350.1/ SM4500-NH3-H) (Electrode – 350.2/ 350.3/SM4500-NH3-F)	120-ml P	0.5mL 1:1 sulfuric acid	28 days
BOD	405.1/SM5210B	500-ml P	None	2 days
Bromide (IC)	300/9056	120-ml P	None	28 days
Chloride (autoanalyzer)	325.2/SM4500-Cl-E	120-ml P	None	28 days
Chloride (IC)	300/9056	120-ml P	None	28 days
Chlorine, total residual	(Titrimetric – 330.2/ 330.3/330.4/SM4500-CL-B) (Colorimetric – 330.5/ SM4500-Cl-G)	250-mL Amb G	None	analyze ASAP
COD	410.1/410.2/SM5220C	120-ml P	0.5mL 1:1 sulfuric acid	28 days
Color	110.1/SM2120B	120-ml P	None	2 days
Cyanide	CLP ILMO 2.1/4.0	250-mL P	4-5 pellets sodium hydroxide	12 days (2)
Cyanide, reactive	SW-846 7.3.3.2/9014	250-mL P	None	14 days
Cyanide, free	ASTM D42982-89	250mL-P	4-5 pellets sodium hydroxide	14 days
Cyanide, weak acid dissociable	SM4500-Cn-I	250mL-P	4-5 pellets sodium hydroxide	14 days
Cyanide, total and amenable to chlorination	335.1/335.2/335.3/335.4/ SM4500-Cn-[C+E+G]/ 9012/9014	250-mL P 500-mL P	4-5 pellets sodium hydroxide 8-10 pellets sodium hydroxide	14 days
Ferrous Iron (colorimetric)	SM3500-Fe-D	250-ml P	1 mL 1:1 hydrochloric acid	analyze ASAP
Fluoride (electrode)	340.2/SM4500-F-C	120-ml P	None	28 days
Fluoride (IC)	300/9056	120-ml P	None	28 days
Hardness (EDTA titration)	130.2/SM2340C	250-ml P	1mL 1:1 sulfuric acid	
Hexane Extractable Materials (HEM)	1664	1-L G (3)	2mL 1:1 sulfuric acid	28 days
Hexavalent chromium	7196/SM3500-Cr-D	250-ml P	None	1 day
Hydrogen ion (pH)	150.1/9040/SM4500-H-B	120-ml P	None	analyze ASAP
Kjeldahl (TKN) and organic nitrogen	351.2	250-ml P	1mL 1:1 sulfuric acid	28 Days

Table 1 - Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Water Matrices

General Chemistry (cont')

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Nitrate – Nitrite	353.2/353.3/ SM4500-NO3-F	120-ml P	0.5mL 1:1 sulfuric acid	28 days
Nitrate (autoanalyzer)	353.2/353.3/ SM4500-NO3-F	120-ml P	None	2 days
Nitrate (IC)	300/9056	120-ml P	None	2 days
Nitrite (colorimetric)	353.2/353.3/ SM4500-NO3-E/ SM4500-NO3-F (w/o Cd reduction)/ 354.1	120-ml P	None	2 days
Nitrite (IC)	300/9056	120-ml P	None	2 days
Nitrocellulose (water)	STL-STL SL SOP	1-L Amb G	None	7 days/40 days(4)
Oil and Grease (gravimetric)	418.2	500-mL or 1-L G (3)	2mL 1:1 sulfuric acid	28 days
Organic Carbon (TOC) (Dohrmann analyzer-Tallahassee)	415.1/SM5310B/SM5310C/ 9060	125-ml Amb G	1mL 1:1 sulfuric acid	28 days
Organic Carbon (TOC) (Shimadzu analyzer-Mobile and Savannah)	415.1/SM5310B/SM5310C/ 9060	125-ml Amb G	1mL 1:1 hydrochloric acid	28 days
Orthophosphate	365.1/365.2/SM4500-P-E	120-ml P	None	2 days
Oxygen, dissolved (electrode)	360.1/SM4500-O-G	BOD Bottle G	None	analyze ASAP
Oxygen, dissolved (Winkler)	360.2/SM4500-O-C	BOD Bottle G	HACH DO reagent powder pillows	analyze ASAP
Phenol, total recoverable (direct)	420.1/9065/SM5530[B+D]	125-mL Amb G	1mL 1:1 sulfuric acid	28 days
Phenols, total recoverable (chloroform extraction)	420.1/9056/ SM5530[B+C+D]	500-ml Amb G	2mL 1:1 sulfuric acid	28 days
Phosphorus, total	365.2/365.3/365.4/ SM4500-P-[B+E]	120-ml P	0.5mL 1:1 sulfuric acid	28 days
Residue, filterable (TDS)	160.1/SM2540C	500-ml P	None	7 days
Residue, non-filterable (TSS)	160.2/SM2540D	500-ml P	None	7 days
Residue, settable	160.5/SM2540F	500-ml P	None	2 days
Residue, total	160.3/SM2540B	500-ml P	None	7 days
Residue, volatile (VS)	160.4/SM2540E	500-ml P	None	7 days
Specific Conductance	120.1/9050/SM2510B	120-ml P	None	28 days
Sulfate (IC)	300/9056	120-ml P	None	28 days
Sulfate (turbidimetric)	375.4/9038/SM4500/504-E	120-ml P	None	28 days
Sulfide	376.1/376.2/SM4500-S2-D/ SM4500-S2-E/9034	500-ml P	2mL 2N zinc acetate	7 days
Sulfide, reactive	SW 7.3.4.2 (9034)	500-ml P	None	14 days
Sulfite	377.1/SM4500-503-B	120-ml P	None	analyze ASAP

Table 1 - Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Water Matrices

General Chemistry (cont')

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Surfactants (MBAS)	425.1/SM5540C	500-ml P	None	2 days
Temperature	170.1/SM2550B	120-ml P	None	analyze ASAP
Total organic halogens (TOX)	450.1/9020/SM5320B	500-ml Amb G	2mL 1:1 sulfuric acid	28 days
Total petroleum hydrocarbons and oil and grease by IR	418.1	125-ml Amb G (3)	2mL 1:1 sulfuric acid	28 days
Turbidity	180.1/SM2130B	120-ml P	None	2 days

Metals

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Ferrous Iron (colorimetric)	SM3500-Fe-D	250-ml P	1 mL 1:1 hydrochloric acid	analyze ASAP
Hexavalent chromium	7196/SM3500-Cr-D	250-ml P	None	1 day
Mercury	245.1/7470	250-ml P or 500-mL P	1mL 1:1 nitric acid 2mL 1:1 nitric acid	28 days
Mercury	CLP ILMO 2.1/4.0	250-ml P or 500-mL P	1mL 1:1 nitric acid 2mL 1:1 nitric acid	26 days (2)
Metals	CLP ILMO 2.1/4.0	250 ml P or 500-mL P	1mL 1:1 nitric acid 2mL 1:1 nitric acid	6 months (2)
Metals (except Hexavalent Chromium and Mercury)	180.1/SM2130B	250 ml P or 500-mL P	1mL 1:1 nitric acid 2mL 1:1 nitric acid	6 months

Volatile Organics – GC

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME (7)
Acrolein & acrylonitrile	603	3 x 40-ml G	None	3 days
Alcohols (DAI)	8015B	3 x 40-ml G	None	14 days
Dissolved Gases in water (FID)	RSK SOP 175	3 x 40-ml G	0.3mL 1:1 hydrochloric acid	14 days
Dissolved Gases in water (TCD)	RSK SOP 175	3 x 40-ml G	None	14 days
Glycols (DAI)	8015B	3 x 40-ml G	None	14 days
GRO/petroleum products	8015B	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	14 days
Halocarbons and Aromatics	502.2, 601/602, 8010/8020, 8021	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	14 days
Methanol and other Solvents(NCASI)	NCASI/STL SL SOP	3 x 40-ml G	None	28 days
Purgeable Aromatic Hydrocarbons	502.2, 602,8020,8021	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	14 days
Purgeable Halocarbons	502.2, 601, 8010, 8021	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	14 days

Table 1 - Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Water Matrices

Volatile Organics – GC (cont')

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Solvents (DAI)	8015B	3 x 40-ml G	None	14 days
Non-Halogenated VOC	8015B	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	14 days

Volatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME (7)
Volatiles	CLP OLCO 2.1/ OLMO 3.2/4.2	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	10 days(2)
Volatiles	524.2, 624, 8240/8260	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	14 days
Volatiles by Isotope Dilution	1624	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	14 days

Semivolatiles by GC

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Benzidines	625,8270C	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Chlorinated Herbicides	515,615/8151	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Chlorinated hydrocarbons	612/8121	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Chlorinated Pesticides	508, 608, 608.1, 608.2, 617, 645, 8080/8081	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Chlorinated pesticides and PCBs as Aroclors	CLP OLMO 3.2/4.2/ OLCO 2.1	2 x 1-L Amb G	None (6)	5 days/40 days (2,4)
Chlorinated pesticides and PCBs as Aroclors	8081/8082	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Haloethers	611/8111	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Microextractables	504/8011	3 x 40-ml G	0.3mL 1:1 hydrochloric acid (6)	28 days
Nitroaniline Pesticides	627	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Nitroaromatics & isophorone	609/8091	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Nitrosamines	607/8071	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Organonitrogen Pesticides	633	2 x 1-L Amb G	None (6)	7days/40 days (4)
Organophosphorous Pesticides	507, 614/614.1, 622/622.1, 8141	2 x 1-L Amb G	None (6)	7 days/40 days (4)
PAHs	610/8100	2 x 1-L Amb G	None (6)	7 days/40 days (4)
PCBs as Aroclors or Congeners	608/8080/8082	2 x 1-L Amb G	None (6)	7 days/40 days (4)

Table 1 - Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Water Matrices

Semivolatiles by GC (cont')

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Pesticides	505, 618	3 x 40-ml G	None (6)	7 days
Petroleum hydrocarbons/EPH/DRO	8015B	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Phenols GC	604,8041	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Phthalate esters	606/8061	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Thiocarbamate Pesticides	634	2 x 1-L Amb G	None (6)	7days/40 days (4)
Triazine Pesticides	619/620	2 x 1-L Amb G	None (6)	7 days/40 days (4)

Semivolatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Base/Neutrals/Acids	525,625/8270	2 x 1-L Amb G	None (6)	14 days/40 days (4)
Base/Neutrals/Acids	CLP OLCO 2.1/OLMO3.2/4.2	2 x 1-L Amb G	None (6)	5 days/40 days (2,4)
Dioxins and Furans	1613/8280/8290	2 x 1-L Amb G	None (6)	30 days/45 days (4)
PCB Congeners	680	2 x 1-L Amb G	None (6)	7 days/40 days (4)
Chlorinated Phenolics	1653	2 x 1-L Amb G	2mL 1:1 sulfuric acid(6)	30 days/30 days (4)
2,3,7,8-TCDD	613	2 x 1-L Amb G	None (6)	7 days/40 days (4)

Table 1 - Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Water Matrices

Liquid Chromatography

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Acrylamide	8316	125 ml Amb G	None	7 days
Acrylic acid	STL SL SOP	40-mL G, TFE septum	None	7 days/ 40 days (4)
Asulam	STL SL SOP	125-ml Amb G	None	21 days (4)
Beniocarb	639	2 x 1-L Amb G	None	7 days/ 40 days (4)
Benomyl (as Carbendazim)	631	2 x 1-L Amb G	None	7 days/ 40 days (4)
Bensulide	636	2 x 1-L Amb G	None	7 days/ 40 days (4)
Bentazon	643	2 x 1-L Amb G	None	7 days/ 40 days (4)
Carbamate & Urea Pesticides	632	2 x 1-L Amb G	None	7 days/ 40 days (4)
Carbonyl Compounds (acetaldehyde and formaldehyde)	8315	2 x 125 ml Amb G	None	3 days/3 days (4)
Cyanizine	629	2x 1-L Amb G	None	7 days/ 40 days (4)
Cyanuric acid	STL SL SOP	125-ml Amb G	None	Not specified
Diquat and Paraquat	549.1	500-mL Amb P or foil wrapped P	sulfuric acid to pH<2	7 days/21 days (4)
Endothall	548	1-L Amb G	None	7 days/ 14 days (4)
Ethylenthiourea	STL SL SOP	125-ml Amb G	None	7 days/ 40 days (4)
Glyphosate	547	125-ml Amb G	None	14 days (18 months if frozen)
Hexachlorophene and Dichlorophene	604.1	2 x 1-L Amb G	None	7 days / 40days (4)
Maleic acid/maleic anhydride	STL SL SOP	40-mL G, TFE septum	None	7 days/ 40 days (4)
Nitroaromatics and Nitramines (explosive residues)	8330	2 x 1-L Amb G	None	7 days/ 40 days (4)
N-Methylcarbamates	8318	2 x 125 ml Amb G	Monocloroacetic acid to pH <3	7 days/ 40 days (4)
N-Methylcarbamoxyloximes & N-Methylcarbamates	531	125-ml Amb G	monochloroacetic acid to pH <3	28 days
Oryzalin	638	2 x 1-L Amb G	None	7 days/ 40 days (4)
PAHs	610/8310	2 x 1-L Amb G	None(6)	7 days/ 40 days (4)
Phthalic acid/phthalic anhydride	STL SL SOP	40-mL G, TFE septum	None	7 days/ 40 days (4)
Picloram	644	2 x 1-L Amb G	None	7 days/ 40 days (4)
Rotenone	635	2 x 1-L Amb G	None	7 days/ 40 days (4)
Thiodiglycol	STL SL SOP	125-ml Amb G	None	7 days/ 40 days (4)

Table 1 - Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Water Matrices

Microbiological

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Bacteria plate count	9215	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodium thiosulfate	6 hours
Chlorophyll	0200H	125-mL Amb G	None	6 months frozen
Coliform, fecal and total (in drinking water)	9222D (Fecal MF) 9222B (Total MF) 9221C (Fecal MPN) 9221B, C (Total MPN)	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodium thiosulfate	30 hours
Coliform, fecal and total	9222D (Fecal MF) 9222B (Total MF) 9221C (Fecal MPN) 9221B, C (Total MPN)	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodium thiosulfate	6 hours
Fecal streptococci	9230C/9230B	2 x 250-mL sterile Nalgene or sterile Whirl-paks	0.2mL 10% sodium thiosulfate	6 hours

Table 2 - Recommended Sample Containers, Preservation, and Hold Time for Parameters Measured in Soil and Solid Matrices

General Chemistry

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Ammonia	350.1/350.3 (EPA-CE)	250-mL P	None	28 days
BOD	EPA-CE : 3-380	500-ml P	None	2 days
Bromide (IC)	300/9056	250-mL P	None	28 days
Chloride (autoanalyzer)	9251/SM4500-Cl-C	250-mL P	None	28 days
Chloride (IC)	300/9056	250-mL P	None	28 days
COD	EPA-CE : 3/373	250-mL P	None	28 days
Cyanide	CLP ILMO 4.0	250-mL P	None	12 days (2)
Cyanide, reactive	SW-846 7.3.3.2/9014	250-mL P	None	14 days
Cyanide, total and amenable to chlorination	9012/9014 (9013)	250-mL P	None	14 days
Extractable organic halogens (EOX)	9023	125-mL Amb G	None	28 days
Fluoride (electrode)	340.2/SM4500-F-C	250-mL P	None	28 days
Fluoride (IC)	300/9056	250-mL P	None	28 days
Hydrogen ion	9045	250-mL P	None	Analyze ASAP
Kjeldahl (TKN) and organic nitrogen	EPA-CE : 3/201	250-mL P	None	28 days
Nitrate (autoanalyzer)	SM4500/NO3-F (EPA-CE : 3-183)	250-mL P	None	28 days
Nitrate (IC)	300/9056	250-mL P	None	28 days
Nitrite (colorimetric)	SM4500/NO3-F (EPA-CE : 3-183)	250-mL P	None	28 days
Nitrite (IC)	300/9056	250-mL P	None	28 days
Nitrocellulose	STL SL SOP	500-mL amb G	None	40days/1 day(4)
Oil and Grease (gravimetric)	9070/413.1 (9071)	250-mL or 500-mL amb G	None	28 days
Organic Carbon	9060	250-mL P	None	28 days
Orthophosphate	365.1/SM4500-P-F	250-mL P	None	28 days
Phenol, total recoverable (direct)	9065	125-mL Amb G	None	28 days
Phenols, total recoverable (chloroform extraction)	9065	125-mL Amb g	None	28 days
Phosphorus, total	EPA-CE : 3-212, 3-213	250-mL P	None	28 days
Residue, total	EPA-CE : 3-58/2540G	250-mL P	None	28 days
Residue, volatile (VSS)	EPA-CE : 3-59/2540G	250-mL P	None	28 days
Specific Conductance	9050	250-mL P	None	28 days
Sulfate (IC)	300/9056	250-mL P	None	28 days
Sulfate (turbidimetric)	375.4/9038	250-mL P	None	28 days

Table 2 - Recommended Sample Containers, Preservation, and Hold Time for Parameters Measured in Soil and Solid Matrices

General Chemistry (cont')

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Sulfide	9034 (9030)	250-mL P	None	28 days
Sulfide, reactive	SW-846 7.3.4.2/9034	250-mL P	None	28 days
Surfactants	425.1/5540C	250-mL P	None	28 days
Total petroleum hydrocarbons and oil and grease by IR	418.1/9070/9073/5520F	250-mL or 500-mL amb G	None	28 days

Metals

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Hexavalent chromium	7196 (3060A)	250-ml P or 500-mL P	None	30 days/ 7 days (5)
Mercury	7471	250-ml P or 500-mL P	None	28 days
Mercury	CLP ILMO 4.0	250-ml P or 500-mL P	None	26 days (2)
Metals	CLP ILMO 4.0	250 ml P or 500-mL P	None	6 months (2)
Metals (except Chromium VI and Mercury)	ICP: 6010 GFAA: 7000	250 ml P or 500-mL P	None	6 months (2)

Volatile Organics – GC

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1, 8)	HOLD TIME (9)
GRO	8015B	3 x 5-g Encore plus 125-mL G Amb	5mL 5% sodium bisulfate solution, methanol, or frozen in water	14 days
Halocarbons and Aromatics	8010/8020, 8021	3 x 5-g Encore plus 125-mL G Amb	5mL 5% sodium bisulfate solution, methanol, or frozen in water	14 days
Purgeable Aromatic Hydrocarbons	8020,8021	3 x 5-g Encore plus 125-mL G Amb	5mL 5% sodium bisulfate solution, methanol, or frozen in water	14 day
Purgeable Halocarbons	8010, 8021	3 x 5-g Encore plus 125-mL G Amb	5mL 5% sodium bisulfate solution, methanol, or frozen in water	14 days
VPH	MA method	3 x 25-g Encore	None	(10)

Table 2 - Recommended Sample Containers, Preservation, and Hold Time for Parameters Measured in Soil and Solid Matrices

Volatile Organics – GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Volatiles	8260/5030	125-mL G Amb	None	14 days
Volatiles	8260/5035	3 x 5-g Encore plus 125-mL G Amb	5mL 5% sodium bisulfate solution, methanol, or frozen in water (8)	48 hrs to preserve/ 14 days(9)
Volatiles	CLP OLMO 3.2	125-mL G Amb	None	10 days (2)
Volatiles	OLMO 3.2/OLMO 4.2	3 x 5-g Encore plus 125-mL G Amb	5mL 5% sodium bisulfate solution, methanol, or frozen in water (8)	48 hrs to preserve(9)/ 10 days(2)

Semivolatiles by GC

PARAMETER	MEHTOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Chlorinated Herbicides	8151	250-ml or 500-mL G	None	14days/40 days (4)
Chlorinated hydrocarbons	8121	250-ml or 500-mL G	None	14days/40 days (4)
Chlorinated Pesticides and PCBs as Aroclors	CLP OLMO 3.2/4.2	250-ml or 500-mL G	None	10days/40 days (2,4)
Chlorinated/ Pesticides	8081	250-ml or 500-mL G	None	14days/40 days (4)
Haloethers	8111	250-ml or 500-mL G	None	14days/40 days (4)
Nitroaromatics & isophorone	8091	250-ml or 500-mL G	None	14days/40 days (4)
Nitrosamines	8071	250-ml or 500-mL G	None	14days/40 days (4)
Organophosphorous Pesticides	8141	250-ml or 500-mL G	None	14days/40 days (4)
PAHs	8100	250-ml or 500-mL G	None	14days/40 days (4)
PCBs	8082	250-ml or 500-mL G	None	14days/40 days (4)
Petroleum hydrocarbons/EPH/ DRO	8015B	250-ml or 500-mL G	None	14days/40 days (4)
Phenols GC	8041	250-ml or 500-mL G	None	14days/40 days (4)
Phthalate esters	8061	250-ml or 500-mL G	None	14days/40 days (4)

Semivolatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Base/Neutrals/Acids	8270	250-mL or 500-mL amb G	None	14 days/40 days (4)
Base/Neutrals/Acids	CLP OLMO 3.2/4.2	250-mL or 500-mL amb G	None	10 days/40 days (2,4)
Dioxins and Furans	8280	250-mL or 500-mL amb G	None	30 days/45 days (4)

Table 2 - Recommended Sample Containers, Preservation, and Hold Time for Parameters Measured in Soil and Solid Matrices

Liquid Chromatography

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Acrylamide	8316	100-mL G	None	14 days
Acrylic acid	STL SL SOP	100-mL G	None	14 days
Asulam	STL SL SOP	100-mL G	None	14 days
Benomyl (as Carbendazim)	631	100-mL G	None	14 days/ 40 days(4)
Carbamate & Urea Pesticides	632	100-mL G	None	14 days/ 40 days(4)
Carbonyl Compounds (acetaldehyde and formaldehyde)	8315	100-mL G	None	3 days/ 3 days(4)
Carbonyl Compounds (formaldehyde)	8315	2 x 125 ml amb G	None	3days/3days(4)
Cyanuric acid	STL SL SOP	100-mL G	None	Not specified
Ethylenthiourea	STL SL SOP	100-mL G	None	14 days
Maleic acid/maleic anhydride	STL SL SOP	100-mL G	None	14 days
Nitroaromatics and Nitramines (explosive residues)	8330	100-mL G	None	14 days
N-Methylcarbamates	8318	100-mL G	None	7 days
Oryzalin	638	100-mL G	None	14 days/ 40 days(4)
PAHs	8310	2 x 1-L amb G	None	14days/40 days(4)
Phthalic acid/phthalic anhydride	STL SL SOP	100-mL G	None	14 days
Thiodiglycol	STL SL SOP	100-mL G	None	14 days

Table 3 - Recommended Sample Media and Containers, Solvent, Preservation, and Hold Time for Parameters Measured in Wipe Matrices

Analyses for Wipe Matrices

PARAMETER	METHOD REFERENCE	ROUTINE MEDIA AND CONTAINER	WIPE SOLVENT & PRESERVATIVE	HOLD TIME
Chlorinated Herbicides	8151/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Methanol / No Preservative	14 days/40 days (4)
Chlorinated Pesticides and PCBs	8081/8082/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Hexane / No Preservative	14 days/40 days (4)
Explosives	8330/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Methanol / No Preservative	14 days/40 days (4)
Mercury	7470/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Acetic acid solution / No Preservative	28 days
Metals (except mercury)	6010/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Acetic acid solution / No Preservative	180 days
PAHs	8100/8270/8310/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Methanol / No Preservative	14 days/ 40 days (4)
Phenols	8041/8270/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Methanol / No Preservative	14 days/ 40 days (4)
Phthalate esters	8061/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Hexane / No Preservative	14 days/ 40 days (4)
Volatiles	8260/STL SL SOP	2" x 2" gauze square / 40 ml G vial with TFE septa	Purge and trap methanol / No Preservative	14 days

Table 4 - Recommended Sample Containers, Chemical Preservation, and Hold Time for Parameters Measured in Solid Hazardous Waste Matrices

Solid Hazardous Waste

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Corrosivity (pH)	9040	250-mL G	None	analyze ASAP
Corrosivity	1110	250-mL G	None	NA
Cyanide, reactive	SW-846 7.3.3.2/9014	250-mL P,G	None	14 days
Ignitability	1010/1030	250-mL G	None	NA
Sulfide, reactive	SW-846 7.3.4.2/9034	250-Ml P,G	None	7 days
SPLP (volatile & nonvolatile fraction)	1312 (6010, 7470, 8081, 8151, 8270, 9012)	500-mL G	None	14 days(11)
SPLP (volatile fraction)	1312 (8260)	250-mL G	None	14 days(11)
TCLP (volatile & nonvolatile fraction)	1311 (6010, 7470, 8081, 8151, 8270)	500-Ml G	None	14 days(11)
TCLP (volatile fraction)	1311 (8260)	250-mL G	None	14 days(11)

Table 5 - Recommended Sample Containers, Chemical Preservative and Hold Time for Parameters Measured in Aqueous Hazardous Waste Matrices

Aqueous Hazardous Waste

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE(1)	HOLD TIME
Base/neutrals/acids	8270	1000-mL G	None	14 days/7 days/40 days (16)
Chlorinated herbicides	8151	1000-mL G	None	14 days/7 days/40 days(16)
Chlorinated pesticides	8081	1000-mL G	None	14 days/7 days/40 days(16)
Mercury	7470	500-mL P	None	14 days/28 days (16)
Metals	6010	500-mL P	None	14 days/180 days(16)
Volatiles	8260	3-40-mL G	None	14 days/14 days(16)

Table 6 - Recommended Sample Media, Preservative, and Hold Time for Parameters Measured in Air Matrices

Volatiles

PARAMETER	METHOD REFERENCE	ROUTINE MEDIA(13, 17)	PRESERVATIVE	HOLD TIME
Volatiles	EPA 18, 0040	Vacusampler	None (Do Not Ice)	30 days
Volatiles and other gases collected in Tedlar Bags	EPA18, 0040	Tedlar Bag	None (Do Not Ice)	3 days

Semivolatiles

PARAMETER	METHOD REFERENCE	ROUTINE MEDIA(13, 17)	PRESERVATIVE	HOLD TIME
Pesticides and PCBs	TO10	PUF Assembly for Low Volume Sampler	None (14)	7 days/40 days
Selected BNA	TO13	PUF/Resin/Filter Assembly for High Volume Sampler	None (14)	7 days/40 days
Selected BNA	TO13	PUF/Resin/Filter Assembly for Low Volume Sampler	None (14)	7 days/40 days
Pesticides and PCBs	TO4	PUF/Filter Assembly for High Volume Sampler	None (14)	7 days/40 days

Table 6 - Recommended Sample Media, Preservative, Hold Time and Sample Minimum Volume Required According to Parameters Measured in Air Matrices

NIOSH/OSHA

PARAMETER	METHOD REFERENCE	ROUTINE MEDIA(13, 17)	PRESERVATIVE	HOLD TIME	SAMPLE MINIMUM VOLUME REQUIRED(15)
Acid anions-Cl, F, NO3, SO4	NIOSH 7903	Silica Gel Sorbent tube	None (14)	21 days	3 liters
Metals by GFAA	OSHA ID-121	Mixed cellulose filter	None	Not specified (180 days)	1250 liters
Metals by ICP	NIOSH 7300/OSHA ID-125	Mixed cellulose filter	None	Not specified (180 days)	1250 liters
Methanol	NIOSH 2000	Silica Gel tube	None (14)	6 Weeks	1 liter
PAH	NIOSH 5515	PUF/Resin/Filter	None Protect from heat and UV light	Not specified (14 days)	200 liters
Aromatic Volatiles	NIOSH 1501 OSHA 07	Charcoal Sorbent tube	None (14)	2 weeks	5 liters
Halogenated Volatiles	NIOSH 1003/ OSHA07	Charcoal Sorbent tube	None (14)	Not specified (14 days)	15 liters

Key to Containers:

P=Plastic; G=Glass; Amb=Amber

Footnote References:

1. Containers should be iced at time of collection in addition to chemical preservation (if applicable).
2. The hold time for CLP methods is measured from the date of receipt in the laboratory.
3. The entire contents of each container must be used for analysis.
4. The first number is the hold time until the extraction; the second time is the hold time for the extract preparation; i.e., the extraction must take place within 7 days of collection and the extract must be analyzed within 40 days of the date of extraction.
5. The digestion must be completed within 30 days and the alkaline digestate must be analyzed within seven days of the digestion.
6. If the sample is chlorinated, sodium thiosulfate or ascorbic acid is added to the vials prior to shipment or is added the time of collection to destroy residual chlorine. If the sample is to also be preserved with acid, ascorbic acid should be used as the dechlorination agent.
7. The hold time for VOC is 7 days if the samples are not preserved with HCl at the time of collection.
8. The sample must be preserved with sodium bisulfate, methanol, or water within 48 hours of collection if not preserved in the field. If the sample contains high levels of carbonates that prevents the use of sodium bisulfate, the sample may be frozen in reagent water within 48 hours of collection.
9. The hold time for VOC is 48 hours if the samples are not preserved with sodium bisulfate, methanol or frozen in analyte-free water within 48 hours of collection.
10. Samples for VPH must be extracted with methanol within 48 hours of collection (1mL methanol per gram of sample). The extract must be analyzed within 28 days of collection.
11. The hold time is the maximum time until the leaching procedure is performed. After leaching, the routine liquid hold times apply for extraction and analysis.
12. If the TCLP or SPLP sample is a liquid or aqueous, a minimum volume of 1L should be supplied to support each analysis. The following are the minimum volumes of liquid sample or leachate to report the target analytes at the LQM limits and below the regulatory threshold limits.
13. Media may be purchased directly from the vendor.
14. No chemical preservative is required. Samples are iced at the time of collection and maintained at method-specified temperature until extraction and analysis.
15. The quantitation limit will be calculated from the volume of sample supplied. The minimum volume for the NIOSH/OSHA methods is the minimum volume that can be sampled to meet the threshold limits. The maximum volume for a sorbent tube is 1.0L (1000mL). This is below "breakthrough" volume of the more volatile gases (e.g., vinyl chloride).
16. The first number is the hold time until the TCLP extraction is performed; the second is the hold time of the leachate extraction or preparation and the third date is the hold time to analyze the leachate or extract.
17. Consumable media provided will be cleaned and leak-checked, when applicable. Shipment of media is billed at the client expense and is supplied contingent on availability. If specified, certified media is available (2-week lead time required) and will be subject to the analytical fees and TAT stipulated in the fee schedule. Blanks can be provided at client request and are billable at the per sample rate. Fees do not include specialized hardware. Contract specific discounts do not apply to consumable media.

7.0 SAMPLE CUSTODY

7.1 Sample Custody Objectives

The primary objective of SL's sample chain-of-custody procedures is to provide accurate, verifiable, and traceable records of sample possession and handling from sample container shipment through laboratory receipt and sample disposition.

Evidence of documentation of sample collection, shipment, laboratory receipt and custody is accomplished utilizing a chain-of-custody record (Figure 7.1). A sample is considered in custody if it is:

- in actual possession of the sampler or transferee
- in view after being in physical possession of the sampler or transferee
- sealed so that sample integrity will be maintained while in possession of the sampler or transferee
- in a secured area, restricted to authorized personnel.

7.1.1 Custody Record Maintenance

Field and laboratory records, including copies of the chain-of-custody forms and associated field documentation, are maintained in a secure area with other project records. All field and laboratory data are reported in bound notebooks and entries are made in waterproof ink. Field and laboratory data entry errors are deleted with a one-line strike through the error. Correction tape or other substances designed to obliterate documentation are strictly prohibited in the laboratory or custody areas. The correction is initialed and dated by the sampling or analytical staff member making the change. Field and laboratory information is documented on prepared forms. All forms for recording field and laboratory data include spaces for data and initials which must be completed by the data recorder. Field and laboratory documentation not recorded on prepared forms is also dated and initialed

7.2 Sample Custody Procedures

All samples are received by the custody technician using custody procedures detailed in STL-SL SOP CU01: *Receipt, Log Number Assignment, and Distribution of Field Samples.* The procedures for the preparation of sampling kits are described in STL-SL SOP CU15: *Preparation of Sampling Containers.*

7.3 Laboratory and Field Custody Procedures

The following procedures apply to the custody activities observed by Savannah Laboratories during sample or legal custody procedures.

7.3.1 Selection and Preparation of Sample Containers Supplied to a Client of Sampling Team

Sample containers provided by STL-SL are constructed from EPA-designated materials and contain EPA-prescribed preservatives. If requested, an STL-SL (Figure 7.2) or client supplied identification label is affixed to the container. A 100-mL plastic container labeled "Container Temperature- For Laboratory Use Only" is pre-filled with tap water and supplied with each sample shipment to monitor sample temperature upon receipt.

Pre-cleaned sample containers are purchased by STL Savannah Laboratories. Containers from each lot are pre-certified in-house prior to use in accordance with STL-SL SOP CU35: *Procedure for Contaminant-Free Containers.*

7.3.2 Chain of Custody Documentation, Traceability, and Sample Integrity

Formal chain-of-custody procedures are initiated by a custody technician who is responsible for organization and relinquishment of sample containers to the client or field personnel.

All field information must be properly recorded on the chain-of-custody form. Proper completion of the form is the responsibility of the field sampling manager or client and is requested prior to the relinquishment of the samples. If the site location is different from the client address, the site location is recorded in the "Project Name" space on the chain-of-custody form or on the right hand side of the form if additional space is required. The sample identities assigned in the field are recorded in the "Sample Identification" column. Common carriers may identify themselves by signing the "Relinquished By" space on the chain-of-custody form.

For samples transported from the field to the laboratory by common carrier, chain of custody is maintained. Completed custody forms must accompany each sealed cooler, and are placed in a plastic bag and taped to the inside lid of the cooler. At the client's request, coolers are sealed in the field with the SL Custody Seal (Figure 7.3) or custody tape by the field sampling team to ensure that tampering will be immediately evident. A unique identification number is recorded on the seal and accompanying chain-of-custody form with waterproof ink.

The custody technician is responsible for the inspection of shipping containers upon laboratory receipt for overall integrity and to ensure that the contents have not been altered or tampered with during transit. If tampering is apparent, the sample receipt custodian immediately contacts the assigned project manager who is responsible for client notification. Any problem or abnormality detected is documented on an Anomaly Report (Figure 7.4), which is completed by the sample custody technician. Any corrective action required by the client is also documented.

If shipping containers arrive intact, they are immediately opened by the custody technician in the receiving area, and the chain-of-custody form and temperature container removed for inspection. Container temperature upon receipt is documented in a bound sample registry (Figure 7.5), or if requested by the client, documented on the chain-of-custody form.

7.3.3 Field Custody

When sample collection is performed by Savannah Laboratories, the SL field sampling manager is responsible for ensuring that chain-of-custody procedures for all sampling events are properly documented. The custody forms and login procedures follow the protocol outlined in Section 7.3.

Prior to field sampling, it is preferable to place waterproof sample labels on each sample container and complete each sample label with as much information as possible in waterproof ink. Field sampling technicians are responsible for ensuring that labels are complete. Each sample is identified in the field by a unique alphanumeric designation on the label.

Adequate sample identification information included on each container label must be included on all field-generated records including: permanent field notebook, individual well log, groundwater elevation form, and chain-of-custody form. This field documentation demonstrates traceability of the containers and samples and links all ancillary records to specific sampling events.

Each sample is packed to ensure against leakage and breakage and to maintain individual sample integrity. All glass containers are secured individually with bubble wrap. All VOA sample vials are wrapped in bubble bags. Plastic bags are supplied by SL to hold ice necessary to maintained the samples at less than 6°C during transit. An attempt should be made by the field sampling team to precool samples to 4°C prior to packing the sample cooler for shipment. Additional information regarding sample containers can be found in Section 6.0 and the appropriate SL SOPs.

When applicable to the site, the following information is documented by the field technicians in the bound field notebook. This field documentation is reviewed, approved, and initialed by the field sampling manager prior to client submission.

Site location
Date/time of sampling
Sample identification (including specific location)
Sample sequence number
Site conditions
Weather conditions
Description of QC samples collected
Names of personnel/visitors
Sampling/purging equipment used
Field analysis data
Field documentation techniques
Well casing composition and diameter
Drilling/boring method
Drilling well type/name
Water table and well depth
Purge volume calculations
Volume of water purged
Date/time of purging
Analytical data to monitor stabilization of well
Use of fuel powered units
Plumbing/tap material construction
Purging flow rate
Purging time
Flow rate at sampling collection
Depth samples taken
Beginning/ending time for composite sampling
Depth soil samples taken
Soil sampling technique used
Type/description of drums
Phases sampled in drums

7.3.4 Sample Documentation, Identification, and Login

A sequential identification number is assigned by division to the project and recorded on the chain-of-custody form, on each sample container submitted with the project and in the bound Sample Registry. Accurate and complete sample documentation must be provided on the chain-of-custody form in order to log samples into the sample registry. The sample registry includes all information necessary to maintain chain of custody including laboratory ID, client (field) ID, and initials of the custody technician. Ancillary information such as sample collection date and requested analyses is transferred from the chain-of-custody form into the LIMS, and appears on the client project-specific acknowledgement.

Once the chain of custody is verified, the project identified by this unique number is logged into the computerized LIMS (Figure 12.1) to transfer the desired work order request to the laboratory. The custody technician checks each sample against the chain-of-custody form for discrepancies between information on the sample label and information provided on the chain-of-custody form. The custody technician also inspects all samples for leakage or obvious seal tampering (if provided). All samples are unpacked in a well ventilated sample receipt area. Face shields are available to each sample receipt staff member for use with any hazardous samples. Samples received in plastic containers which appear to be accumulating or evolving gas are treated cautiously and inspected under a chemical hood because they may contain toxic fumes or be of an explosive nature.

A space labeled "custody intact" provided on the chain-of-custody form is used to describe the sample condition upon receipt. A "Y" indicates no custody problem was identified and a "N" indicates samples or container integrity was compromised and client notification and corrective action is required. At client request, a "Cooler Receipt Form" (Figure 7.6) can be completed to document custodial concerns at sample login.

Discrepancies noted by the custody staff are transmitted to the project and sample manager and are resolved with the client prior to laboratory work assignment. Discrepancies are documented on the Anomaly Report. The project manager and the custody department staff should attempt to resolve custody discrepancies expeditiously to avoid holding time compromises. After a decision concerning a sample has been made, the project manager or sample manager makes an initialed note on the original custody form which states person notified, time, date, and resolution, if applicable. This information is also documented on the Anomaly Report. A faxed or hard copy of custodial resolutions or project order alterations should be secured from the client prior to work initiation. Copies of this documentation are mailed to the client and maintained in the client file.

7.3.5 Sample Preservation

After addition of the project sequential identification number, the samples are distributed to the appropriate laboratory section sample storage areas. Color-coded dots and unique sample bottle types correspond to specific analyses and are stored at designated sample storage areas throughout the laboratory sections. Bound sample storage temperature logs are maintained for all sample storage refrigerators to assure proper temperature maintenance throughout the analytical process.

The color code scheme for the various preservatives used in SL's sample containers is described in the Sample Container Request Form which is submitted to the client along with the sample containers. This two-sided form is shown in Figure 7.7 and 7.8.

When preservation is required, sample containers used by the SL field sampling team contain premeasured portions of preservatives. Preservatives are obtained prior to each sampling event from parent stocks assayed and maintained by the laboratory. The effectiveness of pH adjustment by addition of acid or base to the samples is checked after sampling by pouring a small amount of the preserved sample into a small specimen cup and testing with narrow range pH paper. Because of the risk of compromising sample integrity, VOA samples cannot be checked in the field.

All samples received by Savannah Laboratories are checked for proper pH adjustment by the appropriate preparation or analytical department as soon after receipt as possible. The pH of each sample is checked, documented, and adjusted, if necessary.

7.3.6 Sample Security, Accessibility, Distribution, and Tracking

Only authorized personnel are permitted within the laboratory areas where samples are stored. Sample storage areas are designed to segregate volatile and nonvolatile samples. Standards and extracts are also departmentally controlled and stored separately.

After sample registry login and verification, samples are relinquished from the receiving area to the appropriate sample analysis storage area. Transfer of samples from the sample receipt personnel to the department is documented on the Sample Internal Custody Log (Figure 7.9). Interdivisional sample custody is documented on the Remote Division Sample Internal Custody Log (Figure 7.10). Using LIMS-generated sample preparation worksheets for guidance, samples are extracted, digested, or distilled as appropriate. An example sample preparation log (Wet Chemistry Extraction Log) is shown in Figure 7.11. The extracts, digestates, or distillates are then transferred and relinquished to the appropriate analysis section, where analysis is performed. An example analysis log (Fluoride Analysis) is shown in Figure 7.12. An example of a department-specific tracking form is shown in Figure 7.13 (BNA Extract Custody).

For projects where in-laboratory custody records are required by the client, the project manager and custody department will coordinate the documentation of these records.

Sample holding times are tracked via the LIMS. Sample collection dates are routinely entered into the LIMS with all sample logins. This information allows holding times specific to each department analysis to be tracked by department managers, supervisors, chemists, and analysts through the use of daily status sheets, reference sheets, and preparation worksheets. Date analyzed is recorded via instrument outputs or analysis forms/logs when applicable as an integral part of the raw data. For projects in which reporting the analysis or preparation date is appropriate, the dates are entered into the LIMS.

7.3.7 Sample Disposition Documentation

Upon completion of analytical work, sample custody of unused sample portions, extracts, or digests is relinquished to a central secured storage area. Here the samples, digest, or extracts await disposal, which is performed with the assistance of the LIMS. The LIMS stores clients' specific disposal instructions, compiles results from the analyses of composited samples, prepares sample disposal lists, invoices for disposal and sample return costs, and provides a disposal record for all excess samples.

7.3.8 Interdivisional Custody

The laboratory director at each location monitors the sample load and turnaround time through LIMS-generated reports. If it appears that analysis demand will exceed capacity, samples may be transferred (provided client contracts or arrangements, project QA plans or certification limitations do not prohibit sample transfer) to another SL division to ensure that holding times and turnaround commitments are met. The procedures used by SL are described in SL SOP CU20: *Interlaboratory Sample Exchange* and are summarized below.

If samples are transferred to another division laboratory, full custody is maintained. A completed and signed fax of a Interdivisional Shipping Log (Figure 7.14) is sent to the receiving division custody department. Special LIMS determination codes specific to each laboratory location are utilized to enable the project manager and laboratory director to track sample progress and maintain chain of custody. Copies of the original chain-of-custody form (executed for interdivisional sample submittal), computerized LIMS order information (LOI), and extract or digest preparation logs pertinent to the project order accompany the samples or sample preparations. The accompanying documentation also includes dates of sample preparation and requested analyses. For projects where reporting the preparation or analysis dates/times are entered into the LIMS so that it appears on the final report.

7.4 Verification of Hard Copy Records

Data worksheets, data approval forms, and final reports are routinely printed for verification and signatures. Hard copies of final reports, field data, chain-of-custody forms, and ancillary documentation pertinent to the project will be stored in a secured storage area and placed chronologically within alphabetically arranged client files.

7.5 Facility Security Policy

All external doors are either visually monitored by SL staff or kept locked. Visitors are required to sign in and wear a visitor's badge during their visit and are accompanied at all times by an SL staff member when in the laboratory. Lockable refrigerators and storage cabinets are available for samples requiring this level of security.

Serial Number

STL Savannah Laboratories
A division of Severn Trent Laboratories, Inc.

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

◯ 5102 LaRoche Avenue, Savannah, GA 31404	Phone: (912) 354-7858	Fax: (912) 352-0165
◯ 2846 Industrial Plaza Drive, Tallahassee, FL 32301	Phone: (850) 878-3994	Fax: (850) 878-9504
◯ 900 Lakeside Drive, Mobile, AL 36693	Phone: (334) 666-6633	Fax: (334) 666-6696
◯ 6712 Benjamin Rd., Suite 100, Tampa, FL 33634	Phone: (813) 885-7427	Fax: (813) 885-7049

PROJECT REFERENCE	PROJECT NO.	PROJECT LOCATION (STATE)	MATRIX TYPE	REQUIRED ANALYSES	PAGE ___ OF ___
STL (LAB) PROJECT MANAGER	P.O. NUMBER	CONTRACT NO.			STANDARD REPORT DELIVERY ◯ DATE DUE ______
CLIENT (SITE) PM	CLIENT PHONE	CLIENT FAX			EXPEDITED REPORT DELIVERY (SURCHARGE) ◯ DATE DUE ______
CLIENT NAME	CLIENT EMAIL				NUMBER OF COOLERS SUBMITTED PER SHIPMENT:
CLIENT ADDRESS					
COMPANY CONTRACTING THIS WORK (if applicable)					

SAMPLE DATE	SAMPLE TIME	SAMPLE IDENTIFICATION	COMPOSITE (C) OR GRAB (G) INDICATE	AQUEOUS (WATER)	SOLID OR SEMISOLID	AIR	NONAQUEOUS LIQUID (OIL, SOLVENT, ETC.)	NUMBER OF CONTAINERS SUBMITTED									REMARKS

RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME
RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME

LABORATORY USE ONLY

RECEIVED FOR LABORATORY BY: (SIGNATURE)	DATE	TIME	CUSTODY INTACT YES ◯ NO ◯	CUSTODY SEAL NO.	STL-SL LOG NO.	LABORATORY REMARKS:

FCU036 09.29.99:0

FIGURE 7.1

FIGURE 7.2

Client ____________________

Sample ID ____________________

Location ____________________

Analysis ____________________

Preservative ____________________

Collection Date/Time ____________________

Collected By ____________________

FIGURE 7.3

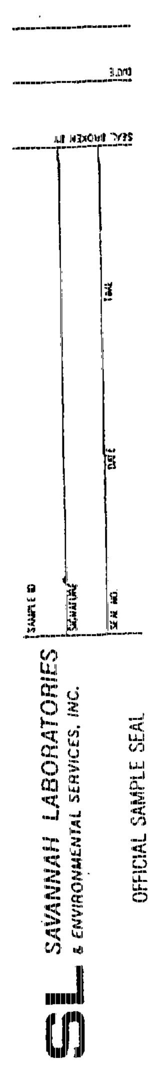
SL SAVANNAH LABORATORIES
& ENVIRONMENTAL SERVICES, INC.
OFFICIAL SAMPLE SEAL
SAMPLE ID
SIGNATURE
SEAL NO.
DATE
TIME
SEAL BROKEN BY
DATE

FIGURE 7.4

ANOMALY REPORT

Date: ______ Log #: ______ Sample ID: ______ Client: ______

Dept: ____ EX ____ GE ____ LC ____ ME ____RA
____CU ____ SG ____ SM ____ VG ____ VM ____AI

Analysis: ______

Reported by: ______

Anomaly:

☐ Sample matrix is different than indicated by log-in.

Logged in as: *Water*, *Soil*, *Oil*

Best described as: *Water*, *Non-aqueous liquid*, *Soil*, *Sludge*, *Oil*, *Product*, *Other* ______

☐ Sample was received with inadequate preservation, and was preserved upon receipt.

☐ Sample received in an incompatible sample container. ____ *glass* ____ *plastic* ____ *other* ______

☐ MS/MSD failed while the LCS/LCSD passed criteria, for a drinking water parameter. Method indicates data flagging.

☐ Target analyte(s) detected in drinking water sample. (Describe below)

☐ Sample exhibits gross non-homogeneity. (Describe below)

☐ Insufficient sample received for analysis.

☐ Data flag may be needed. Discuss with DM/LM before reporting.

☐ Grand Mean exception was utilized for Initial Calibration (specify compounds).

☐ Grand Mean exception was utilized for Continuing Calibration (specify compounds).

Other ______

Custody: *ALWAYS ATTACH A COPY OF COC WITH HIGHLIGHTED DEFICIENCY

☐ Sample description discrepancy between COC & Container

☐ Custody seals broken

☐ Sample container breakage

☐ Incomplete COC

☐ Cooler temp >6°C or frozen

☐ Sample container partially filled

☐ Sample received not listed on COC

☐ Improperly preserved sample

Comments:

Client Notified: ☐Yes ☐No
Contact:

Date:

Resolution:

Route to:
Division PM:

Other Div. PM: SL______ ML______ NL______ FL______ TL______ BL______

FAN038:03.11.99:4

FIGURE 7.5

SL SAMPLE REGISTRY

SL LOG NO.	TIME RECEIVED	DATE RECEIVED	DELIVERED BY	CLIENT ID	NUMBER OF COOLERS RECEIVED	INITIATE ANOMALLY? Y/N	CONTAINER TEMPERATURE CORRECT	ADDITIONAL SAMPLE INFORMATION

FCU007:05.28.99:1

FIGURE 7.6

COOLER RECEIPT FORM

Client:	**Project:**
SL Log #:	**Date Received:**
SL Cooler Receipt Custodian (Signature):	

Use other side of this form to note details concerning custodial discrepancies

		YES	NO
1	Did a shipping slip (air bill, etc.) accompany the cooler shipment?		
2	Were custody seals affixed to the outside of cooler? If YES, enter the following: Seal Identification (if provided):		
3	Were custody seals unbroken and intact at the date and time of arrival?		
4	Were custody papers completed properly (ink, signed, etc.)?		
5	Chain of custody associated with cooler receipt form.		
6	Was wet ice/blue ice used? (Circle which media)		
7	Cooler temperature upon receipt:		
8	*Describe type of packing in cooler (vermiculite, bubble pack, etc.).*		
9	Were sampling containers supplied by SL or client? (Circle which one)		
10	Did all bottles arrive intact and were labels in good condition?		
11	Did all bottle labels agree with custody papers?		
12	Were bubbles present in VOA samples?		
13	Was the project manager notified of any custody discrepancies or excursions?		
14	Was a custody excursion form completed and a copy provided to the project manager? If so, complete No. 15.		
15	Who was contacted? By whom: Date:		

FCL013 02.02.95.1

FIGURE 7.7

STL Savannah

S9102110/SL

DATE/TIME PRINTED: 10.21.99/02:54PM

Shipping Address: Client Name
Street Address
City, State, ZIP Code
Attn: Client Contact

Phone No: 912/354-7858

Account No:

Date of Shipment: 10/20/99

Method of Shipment: UPS

Project Reference: _

Project Site Location: State Location, USA

SAMPLE CONTAINER REQUEST FORM

AQUEOUS			
O	R	LB	PRESERVATIVES
1 L n/m amber glass w/TFE	500 mL m/m plastic	40 mL vial w/TFE	Lab Pk Prep, by: ______ Lab Pk Checked by: ______ Quantity of Lab Pks. Shipped: ______ Proj Mgr: Gloria D. Fulwood Coordinator: Abbie Page Comments: Example Container Request Form
4	2	9	NO. OF CONTAINERS SHIPPED
2	1	3	NO. OF CONTAINERS/SAMPLE
		1	SET(S) OF TRIP BLANKS (3/SET)
SVOC-8270	AS/CR/CU	VOC-8260	ANALYSIS

It is the shipper's responsibility to ensure samples are maintained at the appropriate temperature during transit.

PRESERVATION COLOR CODE KEY

BLACK(BK)	Contains Monochloroacetic Acid. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
RED(R)	CAUTION! STRONG OXIDIZER! CONTAINS NITRIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
GREEN(G)	CAUTION! CONTAINS SULFURIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
BLUE(B)	CAUTION! STRONG CAUSTIC! CONTAINS SODIUM HYDROXIDE. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
WHITE(W)	No preservatives added.
ORANGE(O)	No preservatives added.
TAN(T)	Contains Zinc Acetate. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.
YELLOW(Y)	Contains Sodium Thiosulfate.
LT.BLUE(LB)	CAUTION! CONTAINS HYDROCHLORIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.

DO NOT inhale vapors that may be caused from a chemical reaction between the preservative and sample. Collect sample in a well-ventilated *area or use appropriate breathing apparatus. NEVER RINSE sample containers. If skin contact with preservatives occurs, flush exposed areas* IMMEDIATELY.

FIGURE 7.8

GENERAL SAMPLING INSTRUCTIONS

DO NOT PRE-RINSE CONTAINERS. These containers have been specially prepared for specific analyses (See Preservation Color Code Key). Fill container to within 1" of capacity unless otherwise indicated, cap tightly, label and ice. Some requests require multiple containers to perform all analyses. (See Sample Request Form on reverse side.)

LITER PLASTIC

No preservative n/m:	Physical Properties, Miscellaneous General (BOD)
Red n/m:	Radiological (Rad 226, Rad 228, alpha and beta)

LITER AMBER GLASS

No preservative n/m:	Extractable Organics (BNAs, Pesticides/PCBs, Herbicides), Dioxins/Dibenzofurans

500 ML PLASTIC

Blue n/m:	Cyanide
No preservative m/m:	Physical Properties, Miscellaneous, General
Red m/m:	Metals with Mercury

500 ML GLASS W/TFE

Lt. Blue m/m:	Petroleum Hydrocarbons
Green m/m:	Oil and Grease
Green m/m (amber):	TOX. Fill to capacity.
No preservative w/m: (Nonaqueous)	All Organics (excluding Volatiles), Inorganics, Physical Properties, General

250 ML PLASTIC

No preservative w/m: (Nonaqueous)	Inorganics, Physical Properties, General (single parameter)
No preservative m/m:	Physical Properties, Inorganics (nutrients), Hexavalent Chromium
Red m/m:	Metals without Mercury
Green m/m:	Nitrogen series, Phosphorus
Tan m/m:	Sulfide

250 ML NALGENE

Yellow m/m:	Bacteriological (Coliform, Standard Plate Count) Sterile container - do not touch cap or container interior. Remove Faucet strainer and flush line prior to sample collection.

125 ML AMBER GLASS W/TFE

Lt. Blue m/m:	TOC. Fill to capacity
Green n/m:	Total Recoverable Phenolics
No preservative m/m: (Nonaqueous)	Volatiles. Fill to capacity - no headspace.

120 ML PLASTIC

No preservative m/m:	Physical Properties. Inorganics (single parameter)
Green m/m:	Nutrients. COD (single parameter)

125 ML GLASS

No preservative w/m: (Nonaqueous)	Organics, Inorganics, Physical Properties, General (single parameter)

40 ML GLASS VIAL W/TFE

Lt Blue n/m:	Volatiles (aromatics and/or Halogenated constituents). Fill vials until slightly overflowing with minimum aeration. Place septa W/TFE liner facing sample and seal with NO headspace.
Orange n/m:	EDB, Volatile Halocarbons. Fill as referenced above.
Yellow n/m:	Trihalomethanes (THM). Fill as referenced above.

Container Closure Key (n/m = narrow mouth, m/m = medium mouth, w/m = wide mouth)

CONTAINER SHIPPING INSTRUCTIONS

After sample collection, please check all custody forms and sample containers for discrepancies. Sign the custody form and seal in the enclosed plastic bag. To avoid container leakage during transit, additional plastic bags have been included in the shipment to contain ice for sample preservation. Please place these ice bags between the samples and secure the lab pack for shipment. Return lab packs to Savannah Laboratories & Environmental Services, Inc., 5102 LaRoche Avenue, Savannah, GA 31404. If you have any questions concerning containers shipped or acceptable field substitutions, please contact your project manager or sample coordinator for assistance at (912)354-7878 or FAX (912) 352-0165.

FIGURE 7.9

SAMPLE INTERNAL CUSTODY LOG

SL LOG NO.________________
CLIENT:________________ **COURIER:**________________
 # COOLERS/CLIENT:____________ **TEMPERATURE**________________

GENERAL CONTAINER TYPE LIQUID	P	#
L N/M PLASTIC		
250 AMB GLASS		
500 M/M PLASTIC		
500 M/M AMB GLASS		
250 N/M PLASTIC		
250 N/M PLASTIC		
250 M/M NALGENE		
125 M/M AMB GLASS		
100 M/M PLASTIC		
DO BOTTLE		
SOIL		
250 M/M NALGENE		
OTHER		
PLEASE VERIFY	✓	
TOTAL CONTAINERS		

METALS CONTAINER TYPE LIQUID	P	#
500 M/M PLASTIC		
250 M/M PLASTIC		
100 M/M PLASTIC		
SOIL		
L W/M PLASTIC		
500 W/M PLASTIC		
250 W/M PLASTIC		
OTHER		
PLEASE VERIFY	✓	
TOTAL CONTAINERS		

VOLATILE CONTAINER TYPE LIQUID	P	#
40 ML VIAL		
SOIL		
ENCORE SPLERS/25g		
ENCORE SPLERS/5g		
125 AMB W/M W/SEPTA		
125 AMB W/M GLASS		
AIR		
TEDLAR BAG		
SUMMA CANS		
VACUUM CANS		
TUBES		
OTHER		
PLEASE VERIFY TOTAL CONTAINERS	✓	

EXTRACTION CONTAINER TYPE LIQUID	P	#
L N/M AMB GLASS		
250 M/M AMB GLASS		
500 M/M AM GLASS		
500 M/M PLASTIC		
250 M/M PLASTIC		
SOIL		
L W/M GLASS		
500 W/M GLASS		
250 M/M GLASS		
OTHER		
PLEASE VERIFY	✓	
TOTAL CONTAINERS		

RELINQUISHED INFORMATION:

CUSTODY INITIAL/DATE __________ **CUSTODY** INITIAL/DATE __________ **CUSTODY** INITIAL/DATE __________ **CUSTODY** INITIAL/DATE __________

GENERAL INITIAL/DATE __________ **METALS** INITIAL/DATE __________ **VOLATILES** INITIAL/DATE __________ **EXTRACTION** INITIAL/DATE __________

MISC. BOTTLES STORED IN REFRIGERATOR FOR SUBCONTRACT/REMOTE TRANSFER: ____________________

FIGURE 7.10

REMOTE DIVISION
SAMPLE INTERNAL CUSTODY LOG

SL DIVISION____________________
 # COOLERS____________________
TEMPERATURE(S)____________________

LOG # FOR GENERAL LAB	#	LOG # FOR METALS LAB	#	LOG # FOR VOLATILES LAB	#	LOG # FOR EXTRACTION LAB	#
PLEASE VERIFY ✓		PLEASE VERIFY ✓		PLEASE VERIFY ✓		PLEASE VERIFY ✓	
TOTAL CONTAINERS		TOTAL CONTAINERS		TOTAL CONTAINERS		TOTAL CONTAINERS	

RELINQUISHED INFORMATION:

CUSTODY INITIAL/DATE	**CUSTODY** INITIAL/DATE	**CUSTODY** INITIAL/DATE	**CUSTODY** INITIAL/DATE
__________	__________	__________	__________
GENERAL INITIAL/DATE	**METALS** INITIAL/DATE	**VOLATILES** INITIAL/DATE	**EXTRACTION** INITIAL/DATE
__________	__________	__________	__________

FIGURE 7.11

WET CHEMISTRY EXTRACTION LOG

Job No 329 ____________
Batch ID ____________
Prep Date ____________
Analyst ____________

	Source Lot#	Amt (ml)	TV	Init
MS/MSD				
LCS				

Sample ID / Sample Desription	SDG	Mt	Chemical Name	Amt Exc'd (g)	FV (ml)	D-D	Comments
1. S667877*3/TBS009	F8005	LI	TCL-V-8260-OV			12.29	
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							
13.							
14.							
15.							
16.							
17.							
18.							
19.							
20.							

Method Blank ____________
Lab Control Sample ____________
Matrix Spike ____________
Matrix Spike Duplicate ____________

Assignment Date ____________ Supervisor Initials ____________

FGE221:07.23.99:0-L

FIGURE 7.12

FLUORIDE
Method 340.2/4500-FC

CALIBRATION CURVE		
mg/L	Log (mg/L)	mV
0.1	-1	
0.3	-0.522	
1	0	
2	0.301	
3	0.477	
10	1	
20	1.30	

Date:

Analyst:

Slope:

Correlation Coefficient:

QC Check (Source/True Value) :

Spike (Source/True Value):

Review Approval/Date:

#	SL Log No.	Sample Description	Sample Dilution	Volume or Wet Weight	Sample Potential (mV)	Result (mg/L)	Percent Solids	Result (mg/kg dw)	% REC	% RPD

FGE091A:02.02.98:2

FIGURE 7.13

SEMIVOLATILE EXTRACT CUSTODY LOG

QE #	Total # of Extracts	Box ID	Analysis Required	Date Complete (Final Proj Chk)	Date Relinquished from Extraction Department		Date Received		Instrument Group	Date Disposed
					Int	Date	Int	Date		

FIGURE 7.14

INTERDIVISIONAL SHIPPING LOG
***FAX PRIOR TO SHIPMENT OF SAMPLES**

PM: ____________________

DIVISION SENT FROM: ______________ TO: ________________

DATE OF SHIPMENT: __________________

METHOD OF SHIPMENT: ________________

SL LOG NO. ____________ **DUE:** __________ **LOGGED IN:** ___Y ___N
_____# SAMPLES/MATRIX ________# CONTAINERS
ANALYSIS REQUIRED (DETS PREFERRED) ______________________
____
CERTIFICATION REQUIRED ___Y ___N TYPE ____________________

SL LOG NO. ____________ **DUE:** __________ **LOGGED IN:** ___Y ___N
_____# SAMPLES/MATRIX ________# CONTAINERS
ANALYSIS REQUIRED (DETS PREFERRED) ______________________
____
CERTIFICATION REQUIRED ___Y ___N TYPE ____________________

SL LOG NO. ____________ **DUE:** __________ **LOGGED IN:** ___Y ___N
_____# SAMPLES/MATRIX ________# CONTAINERS
ANALYSIS REQUIRED (DETS PREFERRED) ______________________
____
CERTIFICATION REQUIRED ___Y ___N TYPE ____________________

NOTE: PLEASE PROVIDE DOCUMENTATION OF SPECIAL REQUIREMENTS FOR CERTIFICATION PRIOR TO ANALYSIS.

PM SIGNATURE/DATE: ________________

RECEIVING DIVISION:
SL LOG #'S: ______________________
RECEIPT INITIALS/DATE: ______________________
PROBLEMS ___Y ___N (IF YES, PLEASE DESCRIBE) ______________________
____
FAX INITIALS/DATE: ______________________

8.0 ANALYTICAL PROCEDURES

The ultimate responsibility for analytical method selection lies with the client or regulatory agencies. Whenever possible, laboratory and field analysis of all samples is conducted by EPA approved methodology or guidance. Interpretation of ambiguous or conflicting method requirements is accomplished by consulting with regulatory agencies and EPA Laboratory/QA personnel. When EPA approved methods do not exist or project protocols require alternative methods or modifications of EPA methods (i.e., to achieve lower reporting limits), methods are modified based on scientific logic and regulatory alternative method guidance.

For Gas Chromatographic (GC) methods, which have long lists of targets, have peaks that co-elute, or are subject to matrix interferences (e.g.. Methods 8021, 604/8041, 606/8061, 607/8071, 609/8091, 610/8100, 612/8121), SL recommends the guidance in SW846, which states that GC-Mass Spec (GC/MS) methods are preferred, provided project reporting detection limits are met and costs are not appreciably different. This practice will usually result in fewer false positive detects and more accurate results (usually lower due to the ability by GC/MS to separate unresolved peaks or eliminate matrix interferences).

A detailed SOP has been prepared for each routine analytical method. At a minimum, the SOPs are reviewed annually and updated as needed to include newly promulgated methods. If multiple versions of a reference method exist (e.g., 6010A, 6010B, etc.), the SOP will reflect the different requirements of each version or an SOP will be prepared for each version. Any modifications to the approved methodology are described in the SOPs. Copies of the SOPs are approved by laboratory management and issued under document control. A master copy of each SOP is retained by the laboratory's QA Department and are made available to each staff member involved in the procedure.

When new routine analytes are added to an established procedure or when a new routine method is developed, data for the analyte or method are generated, reviewed, and documented in accordance with SL SOP AN52: *Test Procedure for Method Development and Modification.* The main elements of SOP AN52 are:

-Preparation or modification of an SOP, if required (SL SOP AN01)
-Performance and evaluation of the Initial Demonstration of Capability (SL SOP CA92)
-Performance of an MDL Study (SL SOP CA90)

8.1 Laboratory Glassware

8.1.1 Volumetric Glassware

Savannah Laboratories employs appropriate glassware for all preparatory and analytical operations. For critical measurements, such as standard preparation, Class A volumetric glassware is used when practical. Exceptions include the use of volumetric syringes for volatiles standards preparation and polypropylene volumetric flasks for metals standards.

8.1.2 Glassware Cleaning Procedures

Laboratory glassware washing procedures are adapted from SW-846, *40 CFR Part 136, Standard Methods*, and EPA 600/4-79-109. The procedures are given in STL-SL SOP AN60:*Glassware Cleaning Procedures* and are summarized as follows:

Extractable Organics

The glassware is washed with hot water and a non-phosphate detergent. The glassware is scrubbed vigorously with a brush to remove all artifacts and rinsed three (3) times with tap water. The glassware is allowed to air dry whenever possible and stored inverted or with cap openings covered with aluminum foil or glass stoppers to exclude dust and other contaminants. Whenever possible, precleaned, certified, disposable glassware is utilized for extraction and extract storage.

Volatile Organics

The glassware is washed with tap water and nonphosphate detergent, rinsed thoroughly with organic free water, and oven dried at 110°C-120°C for at least two hours. Class A volumetric glassware is air dried. Glassware is usually stored in the oven until use. Caps and septa are washed in the same manner, but caps are air dried. Highly contaminated glassware is allowed to soak in Nochromix solution overnight, then washed as above. Whenever possible, precleaned, certified disposable glassware is utilized.

General Chemistry, Microbiology, Nutrients, Demands

The glassware is washed with hot tap water and nonphosphate detergent, rinsed thoroughly with tap and deionized water, air dried, and stored inverted or foil placed over cap openings. Bateriological laboratory glassware and collection bottles are autoclave as described in analytical procedures or purchased presterlized (disposable). COD digestion tubes and caps are cleaned with brushing and tap water (no soap) and rinsed thoroughly with deionized water. Tubes for TKN and total phosphorus sample digestions are washed with hot water and phosphate free detergent, and rinsed with tap water, Nochromix, and deionized water. Whenever possible, precleaned, certified disposable glassware is utilized.

Metals

The glassware, plastic, and Teflon items are washed in hot tap water and phosphate-free detergent. They are then rinsed with tap water, 1:1 nitric acid, tap water, and deionized water. For highly contaminated samples, it is recommended that Teflon beakers used for sample digestion are further decontaminated by adding 20mL nitric acid and 12mL hydrochloric acid, covered with a watch glass, and digested on a hot plate for two hours. Following this treatment, they are rinsed with 10% nitric acid and deionized water and allowed to air dry. Whenever possible, precleaned, certified disposable glassware is utilized for digestion and digestate storage.

8.2 Soil Sample Preparation Notes

In the absence of an approved soil method, water methods are adapted for soil matrices. The following soil preparation procedures are applied to parameters in Table 5.2.

1. Fluoride (extractable): Method 340.2/300.0

Approximately 5 g of sample is weighed out exactly and placed in a screw-cap bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle, the supernatant decanted, and the extract is analyzed as a liquid sample.

2. Gross Alpha and Gross Beta Particle Activity: Method 9310

Soil is ground to a fine powder with mortar and pestle, and 50 to 100mg soil is weighed onto a tared planchet. Sample is distributed evenly over planchet surface, fixed with clear acrylic solution, dried, and counted.

3. Chloride (extractable): Method 9251/9252/4500-CL`C/300.0

Approximately 5 g of sample is weighed out exactly and placed in a screw-cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle and the supernatant is decanted. The extract is analyzed as a liquid sample.

4. Sulfate (extractable): Method 9036/9038/375.3/300.0

Approximately 5 g of sample is weighed out exactly and placed in a 100-ml screw cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the extract is filtered using a syringe and filter with a 0.20-um pore size filter and analyzed as a liquid sample.

5. Orthophosphate (extractable): Method 365.1/300.0

Approximately 5 g of sample is weight out exactly and placed in a screw-cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped and placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle and the supernatant us decanted. The extract is analyzed as a liquid sample.

6. Benomyl in Soil: Method 631

Ten grams of sample are extracted and hydrolyzed in 2:1 methylene chloride:acidified methanol. The extract is cleaned by passing through a 5 gram alumina solid phase extraction cartridge. The cleaned extract is then concentrated, filtered, and analyzed as a liquid sample.

7. Acrylamide in Soil: Method 8316

Samples are prepared by adding 10 ml of HPLC grade H2O to 2.0 grams of sample in a 20 mL scintillation vial and sonicating for one hour. The leachate is then passes through a C18 solid phase extraction cartridge. The extract is filtered and analyzed as a liquid.

8.3 Validated Compounds and Modifications of Referenced Analytical Methods

Except for the instances described below, parameters in Tables 5.1 and 5.2 have been determined by the methods referenced with no significant modifications to those methods, other than the use of additional standards for parameters not included in the referenced method lists.

Asulam

Water samples are prepared by adjusting the pH to 3, saturating with NaCl and extracting with 1:1 acetonitrile:ethyl acetate. The extract in injected into the HPLC system following concentration. The HPLC system consists of an isocratic pump and a UV detector.

Thiodiglycol

Water samples are prepared by passing them through a solid phase cartridge. Soil samples are extracted by sonication with calcium chloride solution and then processed through the same cleanup as the water samples. The samples are injected into the HPLC system which consists of an isocratic pump and a UV detector.

Ethylenethiourea

Water samples are analyzed by passing them through a solid phase cartridge. Soil samples are extracted by sonication with water, then processed through the same cleanup as water samples. The samples are injected into the HPLC system which consists of an isocratic pump and a UV detector.

Nitrocellulose

Water samples are filtered through an inorganic membrane filter to isolate the nitrocellulose fibers which are suspended in the sample. The filter is then washed with methanol to remove any sources of organic nitrogen compounds while the nitrocellulose fibers remain on the filter. The fibers are dissolved in acetone, followed by evaporation of the acetone and subsequent hydrolysis of the residue in the presence of sodium hydroxide. Following neutralization, the sample is analyzed for nitrate and nitrite by a cadmium reduction spectrophotometric method or ion chromatography. The mass of nitrogen (as nitrate-nitrite) is used to calculate the concentration of nitrocellulose.

Soil samples are treated with a methanol rinse to remove any interfering sources of nitrogen. Acetone is then added to the soil to dissolve the nitrocellulose fibers. Following the addition of acetone, the sample is sonicated for about 10 hours. Centrifugation is then employed to separate the acetone from the soil residue. The soil residue is then treated and analyzed using the technique described above for water samples.

Phthalic acid/Phthalic anhydride and Maleic acid/Maleic anhydride

Soil samples are extracted by tumbling with reagent water. Soil sample extracts and water samples are adjusted to neutral pH and then cleaned by passing through C8 and amino solid phase extraction cartridges connected in series. Analysis is by HPLC with UV detection. All results are reported as the acid form.

Cryomazine

Cryomazine is extracted from soil samples by sonication with HPLC extraction fluid, consisting of hexanesulfonic acid, triethylamine, ammonium hydroxide, and phosphoric acid. Extract cleanup is accomplished using a C18 solid phase extraction cartridge, followed by HPLC with UV detection analysis.

Resorcinol

Water samples are acidified with phosphoric acid, then passed through a C18 solid phase extraction cartridge and filtered. Analysis is by HPLC using UV detection.

Arsenic in Water by Method 7060 (3020)

Water samples are prepared using the procedures in Section 7 of Method 3020. Arsenic is then determined using the analytical procedures described in Method 7060.

Selenium in Water by Method 7740 (3020)

Water samples are prepared using the procedures described in Section 7 of Method 3020. Arsenic is then determined using the analytical procedures described in Method 7740.

8.4 Reagent Storage and Documentation

Reagents are stored with consideration for safety and maximum shelf life. Storage conditions and documentation maintenance status for various classes of reagents are given in Table 8.1, as well as discussed below.

All acids, except those poured up in small marked containers which are for immediate use, are stored in the original containers in acid storage cabinets.

All bases, except those poured up in small containers for immediate use and those that are standardized for specific purposes, are stored in the original containers within designated areas or storage cabinets.

All flammable solvents, except those poured up for immediate use, are stored in original containers in approved vented flammable storage cabinets which are located indoors.

Dry reagents are stored in designated cabinets in cool, dry areas. Reactive chemicals, cyanides and sulfides are labeled and isolated from other chemicals.

All acids used for metal sample digestions and solvents used for semivolatile sample extraction are tested prior to initial use. Specific acceptable chemical lots are reserved and stored by the vendor(s) and are requisitioned and received as needed by the laboratory. Lot numbers used for digestion or extractions are recorded in bound notebooks in the appropriate departments.

Reagent blanks are analyzed with each sample batch for all methods, validating the purity of all reagents. All reagent containers are dated when received, and dated and initialed when opened (except high use items consumed in less than one week). Documentation is maintained to provide traceability of the reagents used with the analysis of any batch to specific reagent lot numbers.

8.5 Waste Disposal

Savannah Laboratories' divisions operate as either conditionally exempt small quantity generators of small quantity generators of hazardous waste.

All waste disposal is carried out in accordance with Savannah Laboratories' Waste Disposal SOP (CA70). This document includes procedures for identification, storage, personnel training, tracking forms, report forms, safety, as well as details of the disposal.

8.6 Sample Disposal

All waste disposal is carried out in accordance with Savannah Laboratories' Waste Disposal SOP (CA70). This document includes procedures for identification, storage, personnel training, tracking forms, report forms, safety, as well as details of the disposal.

After analysis completion, unused sample portions, extracts, or digests are transferred to a central secured storage area to await disposal. Unless a client requests the project manager to save unused samples, digests, or extracts, disposal from the central storage occurs as soon as holding times have expired or three weeks after results submission.

Requests for extended sample, digest, or extract storage must be provided by the client to the SL project manager in writing (or contract form) prior to sample receipt and extended storage may result in additional fees to be negotiated by the SL project manager prior to sample receipt. SL is not responsible for evaporation or other deterioration of samples, extracts, or digests during extended storage periods.

Samples which are requested to be returned to the client may be picked up at the laboratory by the client, shipped by courier (at the client's expense for packaged shipping) or returned by any other legal means that is arranged by the client. Clients requesting the return of samples should provide detailed shipping instructions.

If a client by contract requires that samples be disposed of by a hazardous waste contractor, the client's name and EPA ID number are used on the manifest and the client is billed for all disposal related costs.

9.0 CALIBRATION PROCEDURES AND FREQUENCY

9.1 Laboratory Equipment

STL Savannah Laboratories is equipped with state-of-the-art instrumentation to provide quality analytical data to clients. A list of the instrumentation maintained by STL Savannah Laboratories is found in Table 9.1. A list of all field instrumentation maintained by the laboratory is contained in Table 9.2.

9.2 Standard Receipt and Traceability

Standards are purchased from commercial sources in stock solutions or mixes designed for the specific methods or as neat analytes. Certificates of analysis are shipped with each standard material by the vendor. When possible, standards are certified to meet or exceed the criteria established by the US EPA or are traceable to NIST standards.

Standard traceability logbooks are maintained by all sections of the laboratory to track the receipt, preparation, and disposition of all standard materials. A lot number is assigned to each standard material and the lot number is documented in the standard traceability logbook along with date of preparation, initials of analyst, concentration, expiration date (if applicable), and solvent (if applicable). If required, a standard preparation narrative is also provided in this logbook to document the preparation steps for each stock standard.

9.3 Standard Sources and Preparation

Savannah Laboratories maintains an inventory of standard materials necessary to calibrate and verify all analytical systems. Table 9.3 lists the standard sources and preparation protocols for various sections of the laboratory. Field instruments requiring calibration standards (conductivity meters and pH meters) use the same sources as laboratory instrumentation. Table 9.4 lists titrants used by the laboratory and information regarding their standardization. Documentation of standard preparation is performed in accordance with SL SOP AN41: *Standard Material Traceability*. This SOP also provides guidance for the extending the expiration date of standards.

9.4 Laboratory Instrument Calibration

The calibration procedures for the various analytical methods are listed below. All CLP protocols are followed as written in the statement of work (SOW).

9.4.1 Metals

Metals are analyzed by three protocols: 200-series are primarily for drinking water and for NPDES compliance; 6000 and 7000-series are primarily for RCRA testing; and CLP protocols are primarily for hazardous waste site monitoring. It should be noted that EPA has promulgated two versions of EPA method 200.7; one for drinking water and the other for NPDES compliance testing.

ICP (Inductively Coupled Plasma-Atomic Emission)

The inductively coupled plasma atomic emission spectrophotometer is standardized daily with a single concentration standard solution containing the metals of interest and a calibration blank. Multi-point calibrations with a minimum of three standards and a calibration blank encompassing the concentration range of interest are analyzed annually and are on file for each ICP. The calibration curve demonstrates the linearity of each metal over the standardization range. Continuing calibration verification (CCV) standards are analyzed after every 10 samples and at the end of the sequence and must meet the acceptance criteria. A calibration blank (ICB or CCB) is analyzed immediately after the verification standards and must meet the acceptance criteria.

After the instrument is standardized, the following calibration verification checks are performed:

Calibration Check	6010	200.7 (NPDES)	200.7 (Drinking Water)
Re-analysis of Standards	+/-5% of true value	+/-5% of true value	+/-5% of true value
Initial Calibration Verification (ICV)	+/-10% of true value	+/-5% of true value	+/-5% of true value
Initial Calibration Blank(ICB)	<RL (<MDL)	<RL (<MDL)	<RL (<MDL)
RL Standard	Detected; +/-50% of true value	Detected; +/-50% of true value	Detected; +/-50% of true value
Interference Check Solutions A and AB	+/-20% of true value	+/-20% of true value	+/-20% of true value
Continuing Calibration Verification (CCV)	+/-10% of true value	+/-5% of true value	+/-10% of true value
Continuing Calibration Blank (CCB)	<RL (<MDL)	<RL (<MDL)	<RL (<MDL)

All sample results must be bracketed by acceptable calibration standards.

AA (Atomic absorption)

Atomic absorption spectrophotometers are calibrated daily with the specified number of calibration standards, including a calibration blank. The correlation coefficient of the regression curve must be greater than or equal to 0.995. An initial calibration verification (ICV) standard is analyzed immediately upon calibration and must meet acceptance criteria. Continuing calibration verification (CCV) standards are analyzed after every 10 samples and at the end of the sequence and must meet the acceptance criteria. A calibration blank (ICB or CCB) is analyzed immediately after the verification standards and must meet the acceptance criteria.

GFAA (Graphite furnace atomic absorption)

Calibration Check	7000-series	200-series	200.9
Minimum number of calibration points	4	4	4
Initial Calibration Verification (ICV)	+/-10% of true value	+/-10% of true value	+/-5% of true value
Initial Calibration Blank(ICB)	<RL (<MDL)	<RL (<MDL)	<RL (<MDL)
Continuing Calibration Verification (CCV)	+/-20% of true value	+/-10% of true value	+/-10% of true value
Continuing Calibration Blank (CCB)	<RL (<MDL)	<RL (<MDL)	<RL (<MDL)

All sample results must be bracketed by acceptable calibration standards.

FLAA (Flame atomic absorption)

Calibration Check	7000-series	200-series
Minimum number of calibration points	4	4
Initial Calibration Verification (ICV)	+/-10% of true value	+/-10% of true value
Initial Calibration Blank(ICB)	<RL (<MDL)	<RL (<MDL)
Continuing Calibration Verification (CCV)	+/-20% of true value	+/-10% of true value
Continuing Calibration Blank (CCB)	<RL (<MDL)	<RL (<MDL)

All sample results must be bracketed by acceptable calibration standards.

CVAA (Cold vapor atomic absorption-Mercury)

Calibration Check	7000-series	200-series
Minimum number of calibration points	6	4
Initial Calibration Verification (ICV)	+/-10% of true value	+/-5% of true value
Initial Calibration Blank(ICB)	<RL (<MDL)	<RL (<MDL)
Continuing Calibration Verification (CCV)	+/-20% of true value	+/-10% of true value
Continuing Calibration Blank (CCB)	<RL (<MDL)	<RL (<MDL)

All sample results must be bracketed by acceptable calibration standards.

9.4.2 General Chemistry

Autoanalyzer

The autoanalyzer is calibrated with a minimum of five calibration standards at least every six months (some procedures/instruments may require daily calibration). The correlation coefficient of the curve must be greater than or equal to 0.995 using a regression fit. Independent calibration verification standards are analyzed immediately following the calibration standards (initial or continuing), after every 10 samples, and at the end of each run. Sample analyses must be bracketed by calibration verification standards that meet control criteria. The calibration curve is verified each day that analyses are performed by the analysis of a midpoint standard and by the analysis of a standard at the reporting limit of the target analyte(s). The standard at the midpoint must be ±10% of the true value of the standard and the standard at the reporting limit must be detected.

Ion Chromatography (IC)

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target analytes with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. Either linear regression or quadratic curve fitting is used, depending on the analyte. The regression correlation coefficient must be greater than or equal to 0.99 for any analyte to be used for quantitation. Calibration verification standards are analyzed immediately upon calibration, after every 10 samples, and at the end of each run. Sample analyses must be bracketed by calibration verification standards that meet the acceptance criteria.

Ultraviolet-Visible (UV-VIS) Spectrophotometer

The spectrophotometer is calibrated with a minimum of five standards at least every six months (some procedures/instruments may require daily calibration). The correlation coefficient of the curve must be greater than or equal to 0.995 using a regression fit. Independent calibration verification standards are analyzed immediately following the calibration standards (initial or continuing), after every 10 samples, and at the end of each run. Data must be bracketed by calibration verification standards that meet control criteria. The calibration curve is verified each day that analyses are performed by the analysis of a standard at the midpoint of the calibration curve and by the analysis of a standard at the reporting limit of the target analyte(s). The standard at the midpoint must be ±10% of the true value of the standard and the standard at the reporting limit must be detected.

Infrared (IR) Spectrophotometer

The infrared spectrophotometer is calibrated with a minimum of five standards at least every six months (some procedures/instruments may require daily calibration). The correlation coefficient of the curve must be greater than or equal to 0.995 using a regression fit. Independent calibration verification standards are analyzed immediately following the calibration standards (initial or continuing), after every 10 samples, and at the end of each run. Data must be bracketed by calibration verification standards that meet control criteria. The calibration curve is verified each day that analyses are performed by the analysis of a standard at the midpoint of the calibration curve and by the analysis of a standard at the reporting limit of the target analyte(s). The standard at the midpoint must be ±10% of the true value of the standard and the standard at the reporting limit must be detected.

Turbidimeter

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. Gelex solid standards are calibrated against formazin standards initially and then quarterly. The instrument is calibrated daily with one Gelex standard for each range of interest. A mid-range calibration verification standard is analyzed for every 10 samples and must meet control criteria in order for bracketed data to be acceptable.

Conductivity Meter

The cell constant of each meter is determined at a minimum annually by the analysis of five KCl standards. To verify the cell constant, a verification standard is analyzed at the beginning of each working day, using a KCl standard in the expected range of the samples. For meters not having automatic temperature compensation, all samples are analyzed at 25 C ± 2 C.

pH Meter

The pH meter is calibrated daily with two standard buffers at pH 7.0 and either 4.0 or 10.0, and checked with a third buffer at 10.0 or 4.0, which must indicate ± 0.10 pH units of its given value. A calibration verification standard is analyzed immediately upon calibration and after every 10 samples. Acceptable calibration verification standards must bracket the sample analyses. Manual or automatic temperature compensation is performed, depending on the meter. Additional checks of the pH meter must be performed with buffers other than 4 or 10 if samples are outside the pH range of 4-10.

Total Organic Carbon (TOC)

The instrument is calibrated according to the manufacturer's recommendations, with a minimum of a single point calibration daily. A calibration verification standard is analyzed immediately upon calibration, after 10 samples, and at the end of each run. Sample analyses must be bracketed by acceptable calibration verification standards.

Ion Selective Electrode (ISE)

Ion selective electrodes are calibrated daily with a minimum of five standards. The calibration curve is established by linear regression applied to the log of the standard concentrations versus potential and must result in a correlation coefficient greater than or equal to 0.995. Calibration verification standards are analyzed immediately upon calibration, after every 10 samples, and at the end of each run. Data must be bracketed by calibration standards that meet control criteria to be acceptable.

Total Organic Halogens (TOX) /Absorbable Organic Halides (AOX)

Although the TOX/AOX instrument provides an "absolute" measurement of halogen, a six-point calibration curve (five point and a calibration blank) is analyzed to confirm the accuracy of the instrument readout. The coefficient of variation (percent relative deviation) must be less than or equal to 20% to confirm the validity of the calibration curve. The TOX/AOX calibration is verified by periodic analysis of a precision and recovery (PAR) standard, a mid-level calibration check standard.

Bomb Calorimeter

The energy equivalent of the bomb calorimeter is determined quarterly by bombing six standard benzoic acid tablets. A control standard is analyzed in duplicate for every batch of samples, and must meet control criteria in order for data to be acceptable.

Dissolved Oxygen (DO) Meter

DO meters are calibrated prior to use either by Winkler titration or the air calibration technique.

Temperature

All laboratory and field thermometers are calibrated annually by comparison with a NIST-certified thermometer. Field meters with automated temperature compensation are checked before use with a calibrated thermometer.

9.4.3 Gas Chromatography (GC)

Volatiles by GC (VG)

Volatile organic compounds (VOC) are analyzed by three protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; and 8000-series are primarily for RCRA testing. These analyses are generally performed using internal standard calibration and quantitation; therefore relative retention time, as defined in the respective SOPs, will be used to determine the identification of the target compounds and bracketing by CCV will not be required unless specified in the method or QAPP. If external standard calibration is used, the absolute retention time window is calculated as three times the standard deviation obtained from a 72-hour sequence or default windows of 0.05 to 0.10 minutes are used for compounds where the calculated window is too restrictive or zero. Bracketing by CCV will be required for external standard calibrations if specified in the method, SOP, or QAPP.

Initial calibration (ICAL) is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard (CCV) must be analyzed periodically as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Calibration Check	500-series(1)	600-series	8000-series
Initial calibration -minimum calibration standards	3 (as the calibration range is extended, the number of points must be increased)	3	5
%RSD criteria (1)	<=20%	<=10%	<=20% (with exceptions noted below)
CCV criteria (%difference or %drift)	+/- 20%	Within Q-table values	+/-15% (with exceptions noted below)
Frequency of CCV	Every 8 hours	Daily	Every 12 hours

(1) Alternatively, a regression curve (linear, quadratic, etc.) may be constructed. If the correlation coefficient of the regression curve is greater than or equal to 0.99, the curve may be used for quantitation of samples.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

8000-series CCV grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Semivolatiles by GC (SG)
Semivolatile organic compounds (SVOC) are analyzed by four protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; 8000-series are primarily for RCRA testing; and the CLP protocols are used for hazardous waste site monitoring. If internal standard calibration is used; relative retention time, as defined in the respective SOPs, will be used to determine the identification of the target compounds and bracketing by CCV will not be required unless specified in the method or QAPP. If external standard calibration is used, the absolute retention time window is calculated as three times the standard deviation obtained from a 72-hour sequence or default windows of 0.05 to 0.10 minutes are used for compounds where the calculated window is too restrictive or zero. Bracketing by CCV will be required for external standard calibrations if specified in the method, SOP, or QAPP.

Initial calibration (ICAL) is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard (CCV) must be analyzed periodically as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Calibration Check	500-series	600-series	8000-series
Initial calibration -minimum calibration standards	3 (as the calibration range is extended, the number of points must be increased)(1)	3	5
%RSD criteria(2)	<=20%	<=10%	<=20% (with exceptions noted below)
CCV criteria (%difference or %drift)	+/- 20%	+/-15% (non-40CFR Methods are +/-10%)	+/-15% (with exceptions noted below)
Frequency of CCV	Every 8 hours	Daily	Every 12 hours

(1) An alternate single point calibration can be performed if the standard response is within 20% of the sample response.
(2) Alternatively, a regression curve may be constructed. If the correlation coefficient of the regression curve is greater than or equal to 0.99, the curve may be used for quantitation of samples.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

9.4.4 Gas Chromatography/Mass Spectrometry (GC/MS)

Volatiles by GC/MS (VM)
Volatile organic compounds (VOC) are analyzed by three protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; 8000-series are primarily for RCRA testing; and the CLP protocols are used for hazard waste site monitoring.

Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable EPA methods. Ion abundance acceptance criteria for VOC tuning with BFB are given below. Mass calibration is performed as an integral part of tuning.

The tune check and calibration check must be performed in the following intervals:

624 - Daily
8260/CLP - every 12 hours.
524.2 - every 8 hours.

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)			
Ion Abundance Criteria			
m/e	**524.2**	**624**	**8260/OLMO4.0(1)**
50	15-40% of mass 95	15-40% of mass 95	8.0-40.0% of mass 95
75	30-80% of mass 95	30-60% of mass 95	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance	Base peak, 100% relative abundance	Base peak, 100% relative abundance
96	5-9% of mass 95	5-9% of mass 95	5.0-9.0% of mass 95
173	< 2% of mass 174	<2% of mass 174	< 2.0% of mass 174
174	> 50% of mass 95	> 50% of mass 95	50-120%% of mass 95
175	5-9% of mass 174	5-9% of mass 174	4.0-9.0% of mass 174
176	> 95% but < 101% of mass 174	> 95% but < 101% of mass 174	93.0-101.0% of mass 174
177	5-9% of mass 176	5-9% of mass 176	5.0-9.0% of mass 176

(1) *8260 criteria taken from CLP OLMO4.0 (January 1998)

Initial calibration (ICAL) is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector.

After the initial calibration standards are injected, a calibration curve is constructed using internal standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD of the calibration curve is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard (CCV) must be analyzed at the required interval as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Volatiles		
Method	**Initial Calibration Check Criteria**	**Continuing Calibration Check Criteria**
624	All targets <= 35% RSD, or alternatively, construct calibration curve	QC Check Sample (20 µg/L) meets limits specified in method –Table 5, Range for Q
8260	CCC <= 30% RSD	CCC <= 20% difference or drift from initial calibration
	SPCC (minimum RF)	
	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	>0.10
	Chlorobenzene	0.30
	1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25-mL purge) (1)
524.2	All targets <= 20% RSD, or alternatively, generate linear, 2nd or 3rd order calibration curve	All targets <= 30% difference from initial calibration, or alternatively, using analyst judgment, all targets must fall on the initial calibration curve

(1) The purging efficiency of 1,1,2,2-tetrachloroethane relative to the internal standard is such that the SPCC criteria cannot be met consistently for a 25mL purge. The response factor is generally in the 0.1 to 0.3 range. The alternate criteria is adopted from the EPA CLP Low Level Statement of Work, a protocol similar in scope and application to SW-846 Method 8260.

SW-846 Method 8260: After the CCC and SPCC are evaluated, all target compounds are evaluated for linearity. If the %RSD is less than or equal to 15%, the average response factor can be used for quantitation. If the %RSD exceeds 15%, a regression curve (linear, quadratic, etc.) may be used for quantitation if the correlation coefficient or coefficient of determination is greater than 0.99.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 15%.

Semivolatile GC/MS (SM)
Semivolatile organic compounds (SVOC) are analyzed by four protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; 8000-series are primarily for RCRA testing; and the CLP protocols are followed for pesticides for hazardous waste site monitoring.

Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable EPA methods. Ion abundance acceptance criteria for SVOC tuning with DFTPP are given below. Mass calibration is performed as an integral part of tuning.

The tune check and calibration check must be performed in the following intervals:

625 - Daily
8270/CLP - every 12 hours.
525.2 - every 8 hours.

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)

	Ion Abundance Criteria		
m/e	525.2	625	8270/OLMO4.0 (1)
51	10-80% of mass 442	30-60% of mass 198	30-80% of mass 198
68	Less than 2% of mass 69	Less than 2% of mass 69	Less than 2.0% of mass 69
69	(reference only)	(reference only)	Present
70	Less than 2% of mass 69	Less than 2% of mass 69	Less than 2.0% of mass 69
127	10-80% of mass 198	40-60% of mass 198	25-75% of mass 198
197	Less than 2% of mass 198	Less than 1% of mass 198	Less than 1% of mass 198
198	Greater than 50% of mass 442	Base peak, 100% relative abundance	Base peak, 100% relative abundance
199	5-9% of mass 198	5-9% of mass 198	5.0-9.0% of mass 198
275	10-60% of mass 442	10-30% of mass 198	10-30% of mass 198
365	Greater than 1% of mass 442	Greater than 1% of mass 198	Greater than 0.75% of mass 198
441	0-100% of mass 443	Present but less than mass 443	Present but less than mass 443
442	Base peak, 100% relative abundance	>40% of mass 198	40-110% of mass 198
443	15-24% of mass 442	17-23% of mass 442	15.0-24.0% of mass 442

(1) *8270 criteria taken from CLP OLMO4.0 (January 1998)

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector.

After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD of the calibration curve is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard must be analyzed daily as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Semivolatiles - GC/MS		
Method	**Initial Calibration Check Criteria**	**Continuing Calibration Check Criteria**
625	All targets <= 35% RSD, or alternatively, construct calibration curve	All targets <=20% difference from initial calibration
8270	CCC <= 30% RSD; SPCC >= 0.050	CCC <= 20% difference from initial calibration SPCC >= 0.050
525	All targets <= 30% RSD, or alternatively, generate linear, 2nd order, or 3rd order calibration curve.	All targets <= 30% difference from initial calibration, or alternatively, using analyst judgment, all analytes must fall on the initial calibration curve

SW-846 Method 8270: After the CCC and SPCC are evaluated, all target compounds are evaluated for linearity. If the %RSD is less than or equal to 15%, the average response factor can be used for quantitation. If the %RSD exceeds 15%, the "grand mean" exception can be applied to the ICAL. Alternatively, a regression curve (linear, quadratic, etc.) may be used for quantitation if the correlation coefficient or coefficient of determination is greater than 0.99.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 15%.

9.4.5 High Performance Liquid Chromatography (LC)

Semivolatile organic compounds (SVOC) are analyzed by three protocols: 500-series are primarily for drinking water; 600-series are primarily for NPDES compliance; and 8000-series are primarily for RCRA testing. If internal standard calibration is used; relative retention time, as defined in the respective SOPs, will be used to determine the identification of the target compounds and bracketing by CCV will not be required unless specified in the method or QAPP. If external standard calibration is used, the absolute retention time window is calculated as three times the standard deviation obtained from a 72-hour sequence or default windows of 0.05 to 0.10 minutes are used for compounds where the calculated window is too restrictive or zero. Bracketing by CCV will be required for external standard calibrations if specified in the method, SOP, or QAPP.

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD of the calibration curve is less than or equal to the acceptance criteria, the average response factor can be used for quantitation. A midpoint calibration verification standard must be analyzed daily as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Calibration Check	500-series	600-series	8000-series
Initial calibration -minimum calibration standards	3 (as the calibration range is extended, the number of points must be increased)(2)	3	5
%RSD criteria(1)	<=20%	<=10%	<=20% (with exceptions noted below)
CCV criteria (%difference or %drift)	+/- 20%	+/-10%	+/-15% (with exceptions noted below)
Frequency of CCV	Every 8 hours	Daily	Every 12 ours

(1) Alternatively, a regression curve (linear, quadratic, etc.) may be constructed. If the correlation coefficient of the regression curve is greater than or equal to 0.99, the curve may be used for quantitation of samples.
(2) An alternate single point calibration can be performed if the standard response is within 20% of the sample response.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

TABLE 9.1
LABORATORY INSTRUMENTS AT EACH SAVANNAH LABORATORIES LOCATION

#	Instrument	Savannah	Tallahassee	Tampa	Mobile
23	PH/ISE Meters	12	4	2	3
11	DO Meters	4	2	3	2
8	Turbidimeters	2	2	2	2
4	TOX/AOX Analyzers	3			1
9	Conductivity Meters	3	2	2	2
1	Bomb Calorimeter	1			
7	Analytical Balances	2	2	1	2
21	Top Loading Balances	7	5	4	5
4	Autoclaves	1	1	1	1
18	Waterbaths	4	7	3	4
8	Biological Incubators	2	2	2	2
7	BOD Incubators	3	1	1	2
26	Drying Ovens	7	9	4	6
11	Block Digestors	5	3	1	2
4	TCLP (Nonvolatile) Extractor Tumblers	SL Custom	SL Custom	SL Custom	SL Custom
4	TCLP (ZHE) Tumblers	2	1		1
11	Sonicators	4	3	2	3
13	Sample Concentrators	4	3	3	3
4	Gel Permeation Chromatographs (GPCs)	2	2		

TABLE 9.2

MAJOR FIELD INSTRUMENTS AT EACH SAVANNAH LABORATORIES LOCATION

#	Instrument	Tallahassee	Savannah	Mobile	Tampa Bay
3	pH/SC/DO/T Meters		1-Corning Checkmate 90		1-Corning Checkmate 90
9	pH/Temp Meters	2-Orion 230A	1-Orion SA-230	3-Orion 23A, 1-Orion 290A	1-Orion 23A
8	Conductivity/ Salinity Meters	1-YSI 33	1-YSI 33	2-YSI 33 1-Orion 120	1-YSI 33
7	DO Meters	1-YSI 51B	1-YSI 51B	1-YSI 50B 1-YSI 51B	2-YSI 50B
4	Turbidimeters	1-Hach 16800	1-DRT 15C	1-Hach 2100P	1-Hach 16800
8	Water Level Meters	1-Slope 51453	1-Fisher	4-Solinst	1-Rocktest Model CPR6
1	Residual Chlorine Colorimeter			1-Hach Pocket Colorimeter	

TABLE 9.3
STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
ICP	Baker/Spex	Stock 1,000 or 10,000 ppm solutions	Room temp	Working std prepped directly from stock	Room temp	Monthly or as needed
AA	Baker/Spex	Stock 1,000 ppm solutions	Room temp	Intermediate stds prepped from stocks. Working stds prepped from intermediates.	Room temp Room temp	Daily Daily
Autoanalyzer	Fisher Baker	Neat material	Room temp	Stock stds prepped from solids. Intermediate stds from stocks. Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed
Ion Chromatograph	Fisher Baker Mallinckrodt	Neat material	Room temp	Stock stds prepped from solids. Intermediate stds from stocks. Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed
UV-VIS Spectrophotometer	Fisher Baker EM	Neat Material	Room temp	Stock stds prepped from solids. Intermediate stds from stocks. Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed

TABLE 9.3
STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
IR Spectrophotometer	Fisher	Neat liquids	Room temp	Stock std prepped from neat liquid. Working stds from stock.	Refrigerator Refrigerator	Monthly Monthly
Turbidimeter	Hach	Standard 4000 ppm formazin solution	Refrigerator	Working stds prepped from stock.	Used immediately	As needed to check Gelex stds
Conductivity Meter	YSI or Fisher	Standard solution or neat KCl	Room temp	Used as is or prepare from neat.	Room temperature	As needed
TOC	Mallinckrodt	Neat KHP	Room temp	Stock std from solid Working std from stock.	Refrigerator Refrigerator	Monthly Monthly
pH Meter	Fisher	Calibration buffer solutions	Room temp	Used as is.	----	----
Ion Selective Electrode (ISE)	Baker	Neat material	Room temp	Stock std from source. Intermediate std from stock. Working std from intermediate.	Refrigerator Refrigerator Used immediately	Monthly Monthly or as needed As needed

TABLE 9.3
STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
TOX	Fisher	Neat material	Room temp	Std from source.	Room temp	Monthly
Bomb Calorimeter	Parr	Neat tablets	Room temp	Used as is.	----	----
GC and GC/MS (Volatiles)	Supelco, Ultra, Accustandard, ChemService, Baxter, Aldrich, Restek, NSI	Neat Solutions (50-5000 ppm)	Freezer	-Stock stds from neat sources. -Intermediate stds from stocks. -Working standards from intermediates and/or purchased solutions.	Freezer Freezer Freezer	-Annually or manufacturer expiration -Semi-annually; monthly for reactive compounds -Weekly
GC and GC/MS (Semivolatiles)	Supelco, Restek, ChemService, Crescent Chemical, Aldrich, Ultra, NSI	Neat Solutions (50-10000 ppm)	Refrigerator	-Stock stds from neat sources. -Intermediate stds from stocks. -Working standards from intermediates.	Refrigerator or freezer Refrigerator or freezer Refrigerator or freezer	Semi-annually or annually as required Semi-annually or annually as required Semiannually or as needed
HPLC	ChemService, Chemical, Supelco Accustandard Radian Absolute	Neat 1000 ppm 1000 ppm 1000 ppm 1000 ppm	Refrigerator	-Stock stds from neat sources. -Intermediate stds from stocks and/or purchased solutions. -Working standards from intermediates.	Refrigerator Refrigerator Refrigerator	Semi-annually Monthly Weekly

TABLE 9.3
STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
Gas Proportional Counter (alpha/beta)	EPA NIST	Sealed Source Stock Soln.	Room temp Metal case Room temp Foil-lined cabinet	Used as is. Working std, prepped from stock.	-- Room temp Foil-lined cabinet	-- As needed
Radon Flask Counter	EPA NIST	Sealed Source Stock Soln.	Room temp Foil-lined cabinet Room temp Foil-lined cabinet	Used as is. Working std, prepped from stock.	-- Room temp Foil-lined cabinet	-- As needed

TABLE 9.4
STANDARDIZATION OF TITRATION SOLUTIONS

Analysis	Solution Requiring Standardization	Standard Identity	Standard Source	Frequency of Standardization
Acidity	Sodium Hydroxide (0.02 N)	KHP	Mallinckrodt	With each batch
Alkalinity	Sulfuric acid	Na_2CO_3	Mallinckrodt	With each batch (or purchased certified)
COD	Ferrous ammonium sulfate	$K_2Cr_2O_7$	Mallinckrodt	With each batch
Chloride	Silver nitrate	NaCl	Baker	With each batch (or purchased certified)
Sulfide	Sulfide working standard	$I_2/Na_2S_2O_3$	VWR/Baker	Weekly
TOC (Soil)	Ferrous sulfate	$K_2Cr_2O_7$	VWR/Baker	With each batch

TABLE 9.5

BALANCE CALIBRATION CHECKS

Analytical Balance	
Class S Weight	**Tolerance**
0.01 g	± 0.0002 g
0.1 g	± 0.0002 g
0.5 g	± 0.0004 g
1 g	± 0.0004 g
10 g	± 0.0005 g
50 g	± 0.0010 g
Top-Loading Balance	
Class S Weight	**Tolerance**
0.1 g	± 0.02 g
0.5 g	± 0.02 g
1 g	± 0.04 g
5 g	± 0.04 g
10 g	± 0.05 g
50 g	± 0.20 g
100 g	± 0.20 g
300 g	± 0.50 g

10.0 PREVENTIVE MAINTENANCE

10.1 Maintenance Schedule

All STL-Savannah Laboratories facilities are equipped with up-to-date computerized instrumentation. In order to gain maximum performance and minimize downtime, regular inspection, maintenance, cleaning, and servicing of all laboratory and field equipment is performed according to the manufacturers' recommendations. A maintenance log is kept for each piece of laboratory and field instrumentation, detailing all maintenance performed on the instrument. Routine repairs and maintenance are performed and documented by the analyst responsible for the particular instrument. Non-routine maintenance is signed and dated by the analyst or repair technician. Routine maintenance procedures for laboratory instrumentation are given in Table 10.1 and each SOP. The frequencies of routine maintenance procedures for Savannah Laboratories' field instrumentation are given in Table 10.2. The service intervals listed in Tables 10.1 and 10.2 are as follows: D=daily; W=weekly; M=monthly; Q=quarterly; SA=semi-annually; A=annually; AN=as needed.

Maintenance contracts are carried for most instrumentation, and close contact is maintained with service personnel to provide optimum instrument functioning.

An extensive spare parts inventory is maintained for routine repairs at the facilities, consisting of GC detectors, AA lamps, fuses, printer heads, flow cells, tubing, certain circuit boards and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be exchanged among the labs.

10.2 Contingency Plan

In general, each facility has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation within each facility, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Savannah Laboratories location (where identical SOPs, QA procedures and instruments are utilized). When shipping samples to another facility, interdivisional chain-of-custody procedures are followed as given in Section 7.

TABLE 10.1								
LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
	D	**W**	**M**	**Q**	**SA**	**A**	**AN**	
ICAP								
Pump Tubing	X							Change.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Quartz Torch							X	Clean and realign.
D-Shaped Mirrors			X					Inspect monthly, clean or replace as needed.
SMITH-HIEFTJE FURNACE AA SPECTROPHOTOMETER								
Sapphire Window	X							Remove and clean.
Flow Rate	X							Check.
Graphite Tube							X	Replace.
Quartz Windows	X							Clean.
Contact Rings and Plates	X							Clean daily, replace if worn.
Filters			X					Inspect monthly, clean or replace as needed.
ZEEMAN FURNACE AA SPECTROPHOTOMETER								
Sampler syringe	X							Check for air daily, flush syringe as needed.
Graphite Tubes	X							Inspect daily, replace as needed.
Graphite Electrodes				X				Inspect quarterly, replace if worn.
Quartz Windows	X							Remove and clean.
LEEMAN PS200 MERCURY ANALYZER AND AUTOSAMPLER								
Pump Tubing	X							Inspect daily, replace as needed.
Standard Cup	X							Inspect daily, replace as needed.
Drying Tube	X							Repack daily at a *minimum*.
Mixing Coil		X						Inspect weekly, clean or replace as needed.
Sample Probe			X					Inspect monthly, clean or replace as needed.
Mercury Lamp							X	Clean or replace.
LEEMAN AP200 PREP STATION								
Autosampler		X						Clean and oil rails.
Tubing					X			Inspect semi-annually, replace as needed.
Bottle Caps				X				Inspect quarterly, replace as needed.
Dispenser				X				Inspect semi-annually, replace as needed.
Water Bath			X					Clean.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
CEM MDS 2100 MICROWAVE								
Pressure Control System	X							Flush.
Cavity and exhaust		X						Clean.
Door			X					Inspect seals and locks.
CONTINUUM FURNACE AA SPECTROPHOTOMETER								
Quartz Windows	X							Remove and clean.
Graphite Tubes	X							Inspect daily, replace as needed.
Contact Rings	X							Clean daily and replace if worn.
Filters			X					Inspect monthly, clean or replace as needed.
D2 Arc Lamp							X	Adjust or replace.
TURBIDIMETER				X				Standardize against formazin.
CONDUCTIVITY METER							X	Replatinize cell when 1 umho/cm range exceeds 90-100%, and when erratic readings cannot be corrected.
pH METER							X	Clean or replace probe.
TOX ANALYZER								
Pyrolysis Tube							X	Clean or replace.
Electrodes							X	Clean.
Electrolytes	X							Replace.
ION CHROMATOGRAPH								
Separator Column				X				Clean.
Guard Column				X				Clean.
Pump Pistons						X		Inspect.
Conductivity Cell						X		Clean.
AUTOANALYZER (TRAACS/LACHAT)								
Pump Platen							X	Replace.
Pump Tubes							X	Replace.
Flow Cell							X	Inspect and clean.
BLOCK DIGESTOR						X		Check calibration.
UV/VIS SPECTROPHOTOMETER					X			Check for wavelength verification.
ION SELECTIVE ELECTRODE							X	Polish electrode.
BOMB CALORIMETER							X	Replace seals.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
AUTOCLAVE								
Pressure Verification	X							Check and document; replace gauge or seals as needed.
Drain Autoclave	X							Visually inspect for leaks and signs of degradation.
Temperature Verification	X							Check with autoclave thermometer; document; replace thermometer as needed.
Cleaning		X						Wash with soapy water; visually inspect for leaks and degradation.
Times				X				Check with stop-watch; replace as needed.
Seals							X	Visually inspect and replace as needed.
TOC ANALYZER								
Catalyst							X	Replace.
Meters (Internal)			X					Check against internal flow meter.
Detector Windows		X						Check and clean.
Humidifiers							X	Fill levels.
Syringe			X					Perform zero point calibration.
GAS CHROMATOGRAPH - SEMIVOLATILES								
Autosampler System							X	Syringe and tubing cleaned. Needles and tubing replace.
Septa	X							Replace as needed.
Column/Injector							X	Change sleeve and cut front of guard column. Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - MASS SPEC SEMIVOLATILES								
Column/Injector							X	Change sleeve and cut front of column.
Septum	X							Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
Splitless Disc							X	Replace.
Autosampler							X	Syringe and tubing cleaned. Needles and tubing replaced.
Rough Pump							X	Oil changed by HP service.
Mass Spectrometer							X	Clean.

TABLE 10.1								
LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
	D	**W**	**M**	**Q**	**SA**	**A**	**AN**	
Tape Head							X	Clean.
Tape Drive							X	Clean.
GAS CHROMATOGRAPH - VOLATILES								
Column							X	Replace.
Septum							X	Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - MASS SPEC VOLATILES								
Column							X	Replace.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
Rough Pump							X	Oil change by HP service.
Gas Manifold	X							Inspect daily, change when pressure reads <500 psi.
Tape Head							X	Clean.
Tape Drive							X	Clean.
GAS CHROMATOGRAPH - SCREENING FOR VOLATILES								
Autosampler							X	Syringe and tubing cleaned. Needles and tubing replaced.
Septa	X							Replace as needed.
Column							X	Replace.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - DAI								
Autosampler							X	Syringe and tubing cleaned. Needles and tubing replaced.
Column							X	Replace.
Septa	X							Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - MASS SPEC AIR ANALYSIS								
TDC Detector								
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Filament	X							Checked for proper temperature when analysis required.
Injector Loop	X	X						Change daily for analysis of atmospheric gases. Change weekly for analysis of non-atmospheric gases.
Column							X	Replace.
Mass Spec								
Cryo-column	X							Inspect for breaks.
Septa		X						Replace as needed.
Column							X	Replace.
Rough Pump							X	Oil change by HP service representative.
Mass Spectrometer							X	Clean.
PURGE AND TRAP								
Sorbent Trap							X	Change.
Purge Flow					X			Inspect semi-annually. Adjust as needed.
HPLC SYSTEMS								
Pumps	X							Pressure check daily, change guard column as needed. Visual leak check daily.
Pump Seals				X				Inspect seals quarterly, replace as needed.
Column	X							Pressure check. Visual leak check.
Detector Fittings	X							Visual leak check.
Detector Optics				X				Remove and inspect filter quarterly, clean or replace as needed.
Autosampler	X							Check seal pack for leaks.
ZYMARK EXTRACT CONCENTRATOR								
Bath		X						Change water and scrub bath; dust outside.
Temperature Verification		X						Verify bath temperature and adjust.
Sensor Diagnostic Test		X						Check each position and adjust.
TENNELEC LB5100								
Sample Change				X				Inspect moving parts quarterly, lubricate as needed.
Detector	X							Inspect for proper operation and response. Serviced by manufacturer only.
Detector gas			X					Inspect monthly, change tank when pressure reads <500 psi. Allow new tanks to dissipate radon for two weeks before use.

TABLE 10.1								
LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
Flow Meter	X							Inspect for proper operation.
BECKMAN LS6500 Liquid Scintillation Counter		X						Inspect for proper operation prior to use. Serviced by manufacturer only.
LUDLUM MEASUREMENTS 2000	X							Inspect for proper operation prior to use. Serviced by manufacturer only.
LUDLUM MEASUREMENTS 182	X							Inspect push rod for high voltage engagement. Inspect instrument noise level without flask.
TCLP EQUIPMENT								
Volatile Rotator			X					Check rotation.
Semivolatiles/Metals Rotator		X						Check rotation.

TABLE 10.2						
FIELD EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE						
EQUIPMENT ITEM	Service Interval					**SERVICE LEVEL**
	D	W	M	Q	A	
TURBIDIMETER HACH 16800/DRT-15C	X					Inspect daily prior to sampling and replace cell as needed.
CONDUCTANCE METER YSI 33	X					Inspect daily, replatinize cell as needed.
pH METER	X					Inspect daily prior to sampling, add filling solution as needed.
CORNING CHECKMATE 90 pH/SC/DO/T° METER	X					Inspect probe, membrane, battery daily prior to sampling. Change parts as needed.
YSI MODEL 50B/51B DISSOLVED OXYGEN METER	X					Inspect probe daily prior to sampling, change as needed.
WATER LEVEL INDICATOR	X					Inspect probe and meter prior to use at every well.

11.0 QUALITY CONTROL CHECKS AND ROUTINES TO ASSESS PRECISION AND ACCURACY AND CALCULATIONS OF METHOD DETECTION LIMITS

The key to a successful QA/QC program is strict adherence to the program during all phases of the project, including pre-sampling discussions; sample collection, preservation, storage and analysis; and validation and reporting of results. Field and laboratory quality control checks, which are part of each sampling trip and laboratory analysis, meet or exceed all agency requirements. Without the proper quality control procedures, analyst and method performance cannot be measured.

When state certification or agency accreditation requirements are non-routine or more stringent than those procedures described below, the requirements are identified according to Sate/Agency Requirement Summaries. These documents outline non-routine analytical quality control practices unique to each program and are derived from administrative codes, regulations, or other similar publications. If project-specific quality assurance plan (QAPP) quality control requirements are more stringent than the general procedures given below, QAPP requirements are followed.

The State/Agency Requirement Summaries are located within each section of the laboratory and are document controlled by the QA Department. Analysts are notified to use the program-specific requirements prior to sample preparation and analysis via status worksheets designated by the Project Manager during project initiation. The State/Agency Requirement Summaries are updated when requirements change.

11.1 Field QC Checks

Savannah Laboratories recommends to their clients that proper control procedures meet or exceed the appropriate regulatory agency field QC requirements.

Blanks, which are collected in the field, are an important link in the quality control data chain for a set of samples. The analytical data derived from these blanks are necessary to assess field-sampling operations. These blanks are used to verify that sample containers, preserving reagents and equipment are contaminant-free. Blanks are also used as a check for potential on-site environmental contamination, to evaluate personnel expertise in sample collection, and to reveal problems that may occur in sample storage and transport.

The field quality control blanks should not be isolated from actual samples. They must be considered as samples and treated identically (preserved with the same reagents, stored and transported in the same containers as the samples, etc.).

The types and frequency of blanks should be included in all quality assurance plans. In cases where data quality objectives dictate more stringent controls, additional field quality control blanks may be required. The following protocol outlines the minimum field blank requirements necessary to assure the validity and integrity of any sampling episode.

Field QC check samples will be analyzed according to the client's instructions and invoiced as samples. Since field QC check samples are usually liquids, they are prepared and analyzed by liquid procedures and reported as liquids. However for batching purposes, unless requested by clients or required by a project specific QA plan, lab QC deliverables are not provided for field QC check samples. Liquid QC samples are batched with soil samples for methods where preparation procedures are the same for both matrices (i.e., volatiles, cyanide, etc.).

11.1.1 Trip Blanks

PURPOSE: The trip blank is to be used when sampling for volatile organics. The purpose is to determine if contamination has occurred as a result of improper sample container cleaning, contaminated blank source water, sample contamination during storage and transportation due to exposure to volatile organics (e.g., gasoline fumes) and other environmental conditions during the sampling event.

PREPARATION: Trip blanks are prepared prior to the sampling event either by the laboratory providing sample containers, or by field team personnel who are responsible for the initial preparation of sample containers and field equipment. The water must be free of volatile organic contaminants. Any appropriate preservatives must be added at the time that the blanks are prepared. The sample containers are sealed, labeled appropriately, and transported to the field in the same sampling kits as the sample vials. These blanks are not to be opened in the field. They are to be transferred to the sample container designated for volatile sample storage, and transported with the samples to the laboratory.

FREQUENCY: One trip blank for each volatile organic analysis (601, 602, 624, 8021, etc.) should be provided per cooler used for storing and transporting volatile sample vials. If a laboratory requires submission of multiple vials for a method, the same number of vials must be submitted for the trip blank.

11.1.2 Field Blanks

PURPOSE: Field blanks are used to evaluate the effects of on-site environmental contaminants, the purity of reagents used as preservatives or additives, and the general sample collection techniques. Field blanks are recommended for all parameters but are not mandatory.

PREPARATION: Field blanks are prepared on-site by filling the sample container(s) with analyte-free water, adding preservatives, sealing the containers and completing the appropriate documentation. The field blanks must be handled in the same manner as the sample group for which it was intended (i.e., blanks must be stored and transported with the sample group).

FREQUENCY: One field blank per parameter group per day or at a frequency of 5% of the samples in the parameter group per day, whichever is greater.

11.1.3 Equipment Blanks

PURPOSE: Equipment blanks are recommended if sampling equipment is field-cleaned. These blanks are used to determine the effectiveness of field cleaning procedures as well as to reveal those sources of contamination that may be found in a trip blank. Equipment blanks must be collected and analyzed for all parameter groups and matrices.

PROCEDURE: The final rinse water (analyte-free) shall be rinsed on or through the sampling equipment, whether pre-cleaned or field cleaned, collected, and placed in appropriate preserved containers. These blanks must be stored and transported with the samples.

FREQUENCY: When less than five samples of a similar matrix are taken, one equipment blank prepared on-site for pre-cleaned or field-cleaned equipment should be collected and analyzed for each parameter. When five to ten samples of a similar matrix are taken, one equipment blank should be collected on field-cleaned equipment or one on-site blank should be collected in pre-cleaned equipment if no equipment is cleaned in the field.

For sampling events involving ten or more samples, a minimum of one blank should be taken on pre-cleaned equipment or at the rate of 5% (whichever is greater) of the samples in each analyte group for all matrices. One blank should be taken on field-cleaned equipment or at the rate of 5% (whichever is greater) of the samples in each analyte group for all matrices.

11.1.4 Field Duplicates

Field duplicates are taken, analyzed, reported and invoiced as required. A minimum of one duplicate for 10% of samples should be taken for all parameter groups and matrices to be collected and analyzed.

11.1.5 Field QC Summary

The recommended frequency of field blanks and duplicates is summarized below:

No. Samples	Pre-cleaned Equipment Blanks	Field-Cleaned Equipment Blanks	Trip Blank (VOCs)	Duplicates
10+	Minimum of one, then 5%	Minimum of one, then 5%	One per cooler	Minimum of one, then 10%
5-9	One*	One*	One per cooler	One
< 5	One*	One*	One per cooler	Not required

* Note: For nine or fewer samples, one equipment blank is recommended from either pre-cleaned or field-cleaned equipment.

If any equipment is cleaned in the field, the blank should be taken from the field-cleaned equipment.

11.2 Laboratory QC Checks

The laboratories employ control samples to assess the validity of the analytical results. Determination of the validity of sample results is based on the acceptance criteria being met by the control samples. The acceptance criteria for each type of control sample are defined in the appropriate SOP. These acceptance criteria are per method requirements or calculated annually from historical data.

Matrix spike results will be utilized for laboratory control when specified by the method. If matrix spikes are out of control, or control is based on laboratory control standards (LCS), then LCS results and method control criteria will ultimately be used to accept or reject the analytical batch. Clients are requested to provide sufficient sample for matrix spikes and are invoiced for matrix spikes.

For CLP protocols or other cases (i.e., when mandated by client or project specific QAPP) where "sample specific" (non-batch) QC is required, matrix spike/duplicate analysis will be conducted on replicate samples provided by the client. In all other cases, matrix spikes will be on a batch-specific basis (not client-, project- or sample-specific basis).

When possible, aliquots for matrix spikes are taken from the same container as the field sample. In some cases with liquid samples, this is not possible, i.e., semivolatile extractables, oil and grease, TPH, etc.

The control samples are analyzed in the same manner as the field samples. QC check samples include the following and are analyzed on an analytical batch frequency unless otherwise stated.

- Quality control check samples are analyzed in duplicate semiannually. These samples are analyzed as blind samples (e.g., WP and WS studies). See section 14.3.1.

- Quality control check standards are analyzed at a frequency equivalent to 5% of the samples in the analytical batch (or at a minimum of one in every 20 samples) in order to verify the analysis.

- Continuing calibration standards are analyzed at a frequency equivalent to 5% of the samples in the analytical batch (or at minimum of one in every 20 samples). Alternatively, the Quality Control check standard may be used to satisfy this requirement. At least one of the checks is a standard at a concentration of 1 - 2 times the laboratory practical quantitation limit (PQL) or reporting limit (RL).

An analytical batch is defined as a group of field samples which are processed as a unit. If the number of field samples in the group is greater than 20, each group of 20 samples or less is handled as a separate batch.

Other QC check samples are analyzed for performance evaluations or as part of internal or external audits as given in Section 14.

If QAPP or agency QC requirements are more stringent than the general procedures given below, QAPP or agency QC requirements are followed.

11.2.1 Organics

Method Blanks: A method blank will be analyzed for each batch of samples.

Lab Control Standards: A blank spike or lab control standard (LCS) will be processed and analyzed (per method requirement) with each batch of samples (except for CLP protocols and other methods which do not require an LCS). For drinking water samples, analyte spike concentrations will be at or near reporting limits as specified for lab-fortified blanks in the 500 series methods. A lab control standard duplicate (LCSD) will be prepared and analyzed if sufficient sample is not supplied for the MS/MSD or duplicate.

Surrogates: Appropriate surrogate(s) (see Tables 5.1 and 5.2) will be added to all samples, standards and blanks.

Matrix Spikes: Matrix spikes will be analyzed at a frequency of 5% of samples. If a method does not specify matrix-spiking compounds, the SW-846 or CLP matrix spiking compounds will be used. Appropriate matrix spikes will be used for other chromatographic methods in which matrix spikes are not defined. Matrix spikes containing all method-specified compounds should be analyzed monthly to generate accuracy and precision limits.

Matrix Spike Duplicates/Sample Duplicates: Duplicate samples or matrix spike duplicates will be analyzed at a frequency of 5% of samples. In cases where duplicate matrix spikes are used, precision data are obtained on only the matrix spiking compounds.

NOTE: Unless requested by the client, matrix spikes are not routinely performed on TCLP, SPLP, EPTOX., or waste dilutions.

Full List Spikes: For projects that require spiking a laboratory control sample (LCS) or matrix spike (MS) with all target compounds, re-extraction and/or re-analysis of the samples in the batch will not be performed if :

1) The recoveries of no more than one (1) compound when 5 to 10 compounds are spiked, two (2) compounds when 11 to 20 compounds are spiked, three (3) compounds when 21-30 compounds are spiked, or five (5) compounds when more than 30 compounds are spiked are determined to be outside the control limits, and recoveries for all spiked compounds are positive. When <5 compounds are spiked, all compounds should be within control limits.

2) The recovery of a spike exceeds the upper control limit (UCL) and the compound is not detected in any sample in the analytical batch.

As indicated in Methods 8260 and 8270, the following compounds have erratic recoveries under the routine conditions of the preparation and analytical procedures and will not be evaluated for corrective action nor included in the count (1) above if included in the LCS or MS:

VOC: Acrolein
Benzyl chloride
Carbon disulfide
2-chloroethyl vinyl ether
Pentachloroethane

SVOC: Aniline
Benzidine
Benzoic acid
Hexachlorocyclopentadiene
Hexachlorophene
Kepone
Alpha, alpha-Dimethylphenethylamine
Methapyrilene
4,4-Methylbis(2-chloroaniline)
p-Phenylenediamine

The above guidance is used as the default for evaluation of full target spikes in organic analyses unless other corrective actions are defined in a project-specific quality assurance plan or in an SL pre-project plan.

11.2.2 Inorganic and General Chemistry

Calibration Blanks: Calibration blanks are non-digested blanks which are analyzed at a frequency of 10% of samples.

Method Blanks: Method blanks should be processed and analyzed with each batch of samples of the same matrix.

Lab Control Standards: A blank spike or lab control standard will be processed and analyzed with each batch of samples (except for CLP protocols and other methods which do not require an LCS). A lab control standard duplicate (LCSD) will be prepared and analyzed if sufficient sample is not supplied for the MS/MSD or duplicate.

Matrix Spikes: Matrix spikes will be analyzed at a frequency of 5% of samples.

Matrix Spike Duplicates/Sample Duplicates: Duplicate samples or duplicate matrix spikes will be analyzed at a frequency of 5% of samples.

NOTE: Unless requested by the client, matrix spikes are not routinely performed on TCLP, SPLP, EPTOX., or waste dilutions.

11.2.3 Microbiology

Quality control checks are routinely performed for all microbiological analyses. Strict requirements for the lab-generated deionized water must be met before it can be used in any testing. Each monitored parameter, its monitoring frequency, and its acceptance limits is as follows: residual chlorine, monthly, < 1.0 mg/L; trace metals (total Cd, Cr, Cu, Ni, Pb, Zn), annually, < 1.0 mg/L, individual metals < 0.05 mg/L; conductivity, daily < 2.0 umho/cm; heterotrophic plate count, monthly, < 500 CFU/mL; inhibitory residue, annually or for each new lot of detergent, less than 15% difference between groups; suitability, annually, ratio between 0.8 and 3.0.

Other laboratory QC practices are utilized to provide accurate microbiological results. Positive and negative microbiological controls are run with each new lot of medium. Autoclave tape is used to ensure proper sterilization of sample containers, media, etc. Incubators are maintained at 35 ± 0.5° C and water baths at 44.5 ± 0.2° C. Thermometers used for these monitoring purposes are calibrated annually against an NIST-certified thermometer. Other equipment, such as the dissecting microscope and colony counter, is maintained in clean operating condition at all times.

Microbiological samples are analyzed in duplicate at a rate of 10% of positive samples. A positive control sample is analyzed with each batch of coliform samples. A negative control is analyzed at least monthly. Additionally, all drinking water samples positive for total coliform must be confirmed. For environmental samples, 10% of samples positive for total coliform must be confirmed. A completed test for MPN analysis must be performed on 10% of all confirmed samples or at least quarterly.

Blanks are routinely analyzed with microbiological samples. For membrane filter analyses, sterile dilution water blank is run initially, after every 10 samples, and at the end of each analytical run. For MPN analysis, sterile dilution water is added to a lauryl tryptose broth tube for a blank for each analytical run.

11.2.4 Radiochemistry

Background Count: Background counts are obtained at a frequency of once per day for gross alpha, gross beta and radium 228; and determined for each flask prior to sample introduction for radium 226.

Method Blanks: Method blanks are analyzed at a frequency of 5% of samples of the same matrix.

Lab Control Standards: Lab control standards are analyzed with each batch of 20 samples of the same matrix.

Matrix Spike/Matrix Spike Duplicate or Sample Duplicates: These are analyzed with each batch of 20 samples of the same matrix.

11.3 Routine Methods Used to Assess Precision and Accuracy

A system for assessing precision and accuracy through tabulation (manual or electronic) is initiated for each parameter upon method validation. Control calculations are based on procedures in *The Handbook for Analytical Quality Control in Water and Wastewater Laboratories* (EPA, 1979) and contain both "warning limits" (± 2 standard deviations) control charts and "control limits" (± 3 standard deviations). Control limits are updated annually for all parameters. A minimum of ten data points is used to update these limits. Formulas used for calculations of precision and accuracy are provided in Section 5.0.

11.4 Method Detection Limits and Reporting Limits

Method detection limits (MDLs) are determined annually in accordance with the procedures in SW-846 and Appendix B of 40 CFR Part 136. This procedure includes analyzing seven or more (40 CFR Part 136) or a minimum of three (SW-846 Chapter One) prepared spikes or standards in reagent water at levels 3-5 times the estimated detection limit. The standard deviation of the replicate measurements is calculated, and the MDL is computed by multiplying by the appropriate Student's t value (n-1 degrees of freedom) for the appropriate 99% confidence level (for seven replicates, t = 3.14).

The MDL calculated by the procedure described above is defined as the minimum concentration of a substance that can be measured in reagent water and reported with a given confidence that the analyte concentration is greater than zero. SL makes no claim that the MDLs determined by this statistical procedure are obtainable in environmental samples.

For other protocols (i.e., Contract Laboratory), other procedures are used to estimate detection limits.

Since MDLs are based on the analyses of standards in reagent water, they may not be useful in reporting data for environmental samples; therefore, practical quantitation limits (PQL) or reporting limits (RL) are typically used for reporting a non-detected parameter. Reporting limits are defined as the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions and are determined to be the lowest concentration standard or the sample equivalent of the lowest concentration standard in the initial calibration.

The method detection limits and reporting limits are determined annually by the Corporate QA Manager in conjunction with Corporate Management and the division laboratory directors, laboratory managers, and QA Officers, from the data submitted by the four Savannah Laboratories' divisions.

Reporting limits for radiological analyses are from recommended values in EPA 40 CFR Chapter 1 (7-1-93 Edition) Section 141.

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12.0 DATA REDUCTION, REVIEW, AND REPORTING

12.1 Introduction

In order to provide the highest quality data possible, an extensive system for sample custody, data reduction, review, and reporting has been implemented.

12.2 Sample Custody

Upon receipt of the samples, the custody forms are checked against the sample identifications listed on the containers by the custody technicians, and a unique SL log number is assigned to each sample group. Any discrepancies are noted, including cooler temperatures, broken bottles and/or misidentified samples. Clients should be immediately notified if discrepancies exist.

After receipt, the samples are delivered to the appropriate laboratory sections where the samples are checked for proper preservation and this information is recorded in bound notebooks when applicable. When necessary, the samples are then stored in refrigerators that are monitored for temperature.

12.3 Organization and Initiation of Sample Analyses

The key to Savannah Laboratories' sample flow, analysis, data/QA review, archiving, and reporting system is the single LIMS network which controls the day to day production of the laboratories. This system, which is summarized in the figure entitled *Data Tracking and Submittal*, provides project managers, QA personnel, and all analysts immediate information on the status of any sample in all five facilities. This system schedules and prioritizes all work, provides a mechanism for sample tracking, review of sample results and QC data, generation of reports and invoices, and archiving of all reports and associated QC data. The policies and procedures for the LIMS and other computer systems are described in the current revision of the Savannah Laboratories' *Software Quality Assurance Plan*.

Upon receipt of custody forms, the project manager instructs data management personnel to log the sample analysis request and identification into the LIMS. The LIMS is based on an ADDS Mentor 7000 computer (NCR) which links the laboratories via telephone multiplex. This enables any project manager, section manager, QA manager, laboratory director, or analyst with authority to access and check the status of all projects.

If special handling or data packaging is required, the QA department and the laboratory receive copies of the custody forms and computer acknowledgments or a pre-project plan. A sample delivery group (SDG) sheet is established and distributed to all affected departments including the various laboratory analysts, project managers, and section managers.

After the sample analysis request is logged into the LIMS and approved, the LIMS generates worksheets which are printed and distributed.

12.4 Sample Analysis and Data Reduction

Through the use of the worksheets and/or SDG sheets, the samples are prepared following the procedures given in each of EPA's approved methods. The preparation information is recorded in signed notebooks throughout the laboratory.

12.4.1 Data Reduction

Most sample concentration results are read directly from instrumentation without further reduction or calculations. Dilution factors are applied upon the dilution of samples having concentrations above the calibration range.

In many cases, these are input into the instrument computer and correct results are calculated automatically. In other cases, a manual calculation may be made. Soil/solid waste concentration results for all laboratory sections are calculated on a dry weight basis, prior to reporting, by dividing the instrument result by the fractional dry weight.

Other than the cases discussed above, data obtained by the following method/instrument are directly reportable: GC, GC/MS, metals, general chemistry automated colorimetry, TOC, DO, turbidity, and pH.

Data from methods requiring reduction prior to reporting include titrimetric methods, BOD, COD, conductivity, manual UV/VIS/IR, residue, TOX, and radiochemical parameters.

Table 12.1 gives equations used in computer-controlled instrumentation for data reduction as well as equations used for the manual calculation of reportable concentration results.

The laboratory raw data containing the instrument-generated reports, manually calculated results, and all supporting preparation, calibration, and analytical data are retained at the individual work stations until reports are issued unless additional handling or data packaging is required.

All pH and conductivity meters should be temperature compensated. Cell constants for field conductivity meters are determined by laboratory personnel annually as given in Section 9.4.2. Field conductivity is calculated as given in Table 12.1. All other field data are read directly from instrumentation.

Bound field notebooks are used for documentation of required data reduction. Calculations are recorded in waterproof ink.

When data are reported from dual columns (e.g., gas chromatography), the default procedure of STL Savannah Laboratories is to report the highest result between the primary and confirmation columns if the relative percent difference (%RPD) is <40%. If the %RPD exceeds 40%, the analyst evaluates the data for the presence of matrix interfemeces and reports the result that is most appropriate for that sample and flags the result to note the discrepancy.

12.4.2 Chromatographic and Data File Identification

Chromatograms and data files are given a unique alphanumeric identification by the chemists initiating the analyses in each section where appropriate. These file identification numbers reflect either the date the sequence was initiated (GC sections), the order in which the samples were analyzed (GC/MS sections), and/or the sample identification and log numbers given by the client and listed on the LIMS.

SAMPLE TRACKING and DATA SUBMITTAL

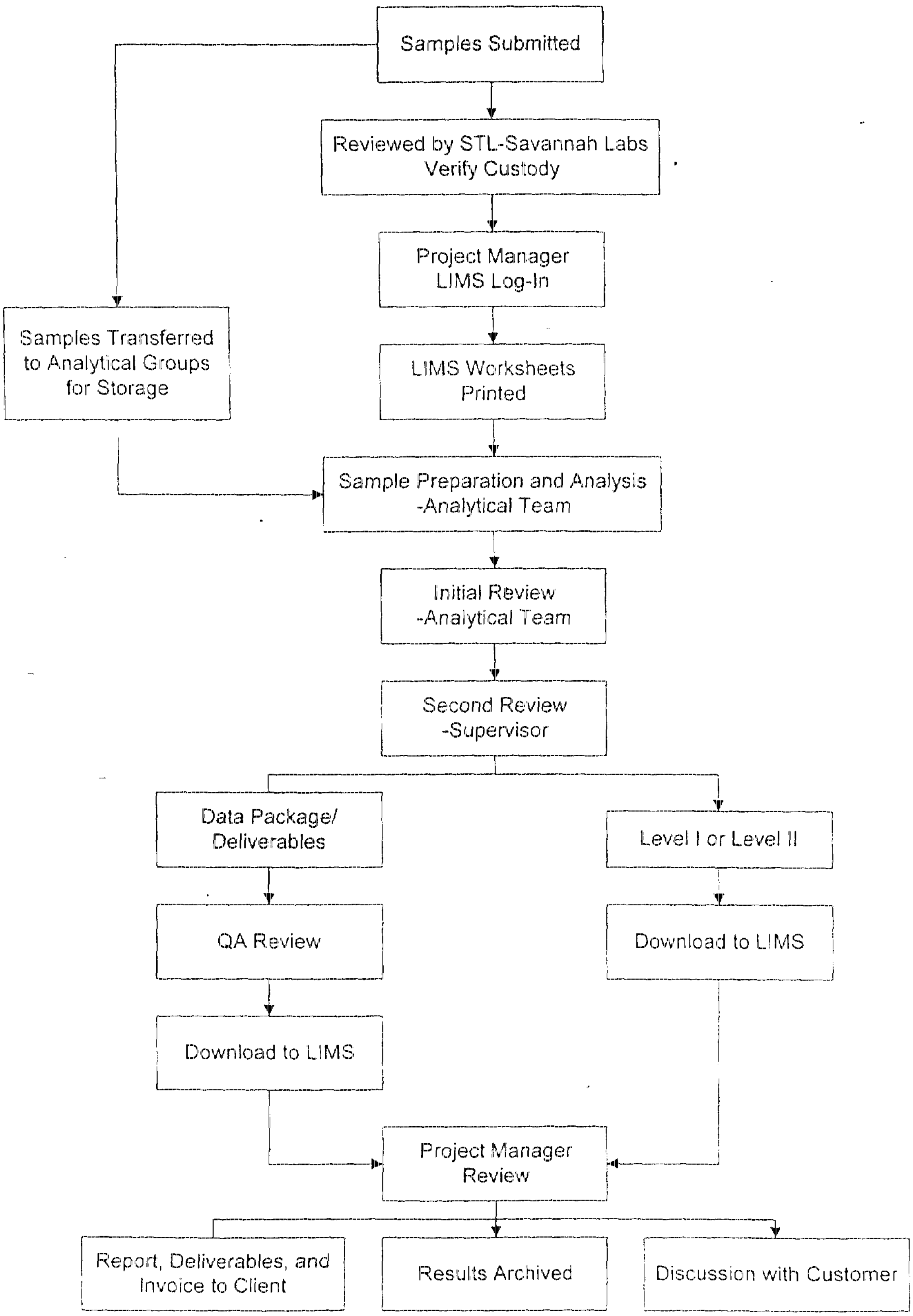

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporting Units	
BN/A Extractables by GC/MS [Internal Standard Method (625 and 8270)]	**Liquid**	**Solid**
$$RRF_s = \frac{A_S}{A_{IS}} \times \frac{C_{IS}}{C_S}$$ RRFs = relative response factor of standard A_S = area of standard (area counts) A_{IS} = area of internal standard (area counts) C_{IS} = concentration of internal standard (ug/ml) C_S = concentration of standard (ug/ml) $$Water\ conc\ (ug/L) = \frac{A_S}{A_{IS}} \times \frac{C_{IS}}{RRF_s} \times \frac{V_f}{V_i} \times DF$$ V_f = final extract volume (ml) V_i = initial sample volume extracted (L) DF = dilution factor $$Soil\ conc\ (ug/kg) = \frac{A_S}{A_{IS}} \times \frac{C_{IS}}{RRF_s} \times \frac{V_f}{W_{spl}} \times \frac{1}{(\%solids \times 0.01)} \times DF$$ W_{spl} = weight of sample extracted	ug/L (or mg/L)	ug/kg (or mg/Kg)

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporting Units: Liquid	Reporting Units: Solid
VOCs by GC/MS and GC	**Liquid**	**Solid**
$$Relative\ Response\ Factor\ (RRFs) = \frac{A_S}{A_{IS}} \times \frac{W_{IS}}{W_S}$$ A_S = area of standard (area counts) A_{IS} = area of internal standard (area counts) W_{IS} = weight of internal standard (ug) W_S = weight of standard (ug) $$Water\ conc\ (ug/L) = \frac{A_S}{A_{IS}} \times \frac{W_{IS}}{RRF_s} \times \frac{DF}{V_{spl}}$$ DF = dilution factor V_{spl} = sample volume purged $$Soil\ conc\ (ug/kg-dw) = \frac{AS}{AIS} \times \frac{WIS}{RRFs} \times \frac{DF}{Wspl} \times \frac{1}{(\%solids \times 0.01)}$$ Wspl = weight of sample purged (Kg)	ug/L (or mg/L)	ug/kg (or mg/Kg)
Pesticides/PCBs and Other GC Procedures	**Liquid**	**Solid**
$$Response\ Factor\ (RFs) = \frac{ug\ of\ standard}{peak\ area}$$ $$Water\ conc.(ug/L) = RF_s \times peak\ area \times \frac{V_f}{V_i} \times \frac{DF}{V_j}$$ V_i = initial sample volume (L) V_f = final extract volume (ml) V_j = injection volume (ml) DF = dilution factor $$Sediment\ conc\ (ug/kg-dw) = RF \times peak\ area \times \frac{V_f}{(W_{spl})(\%\ solids \times 0.01)(V_j)}$$ W_{spl} = weight of sample extracted (Kg)	ug/L (or mg/L)	ug/Kg (or mg/Kg)

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS		
Equations	**Reporting Units**	
Metals	**Liquid**	**Solid**
$$Water\ conc\,(mg/L) = \frac{y-b}{m} \times \frac{V_f}{V_i} \times DF$$ y = $absorbance_{slp}$ b = y intercept from calibration curve (absorbance) m = slope from calibration curve [absorbance/(mg/ml)] V_f = final digest volume (mL) V_i = volume of sample (L) DF = dilution factor $$Soil\ conc\,(mg/kg-dw) = \frac{y-b}{m} \times \frac{V_f}{W_{spl}} \times \frac{1}{(\%solids \times 0.01)} \times DF$$ W_{spl} = weight of sample (Kg)	ug/L (or mg/L) mg/L (or ug/L)	ug/Kg (or mg/Kg) mg/Kg (or ug/Kg)
UV/VIS and IR Procedures	**Liquid**	**Solid**
$$Water\ conc\,(mg/L) = \frac{y-b}{m} \times \frac{V_f}{V_i} \times DF$$ y = $absorbance_{slp}$ b = y intercept from calibration curve (absorbance) m = slope from calibration curve [absorbance/(mg/ml)] V_f = final digest volume (mL) V_i = volume of sample (L) DF = dilution factor $$Soil\ conc\,(mg/kg-dw) = \frac{y-b}{m} \times \frac{V_f}{W_{spl}} \times \frac{1}{(\%solids \times 0.01)} \times DF$$ W_{spl} = weight of sample (Kg)	mg/L	mg/Kg

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS		
Equations	**Reporting Units**	
General Titrimetric Procedures	**Liquid**	
$$Analyte, mg/L = \frac{N_{titrant} \times Titer}{Vol. of\ sample\ titrated} \times eq. wt. \times 1000$$	mg/L	
BOD	**Liquid**	
$$BOD, mg/L = \frac{(Int. DO - Final\ DO) - Seed\ Correction\ Factor}{Vol.\ fraction\ of\ sample}$$	mg/L	
COD	**Liquid**	
$$COD, mg/L = \frac{(Blk\ titer - sample\ titer) \times NFAS \times 8000}{Vol. of\ sample, mL}$$	mg/L	
Conductivity	**Liquid**	
$$Cell\ cons \tan t = \frac{1000}{Observed\ conductivity\ of\ 1000 - \mu S/cm\ std.}$$	μS/cm	
Residue	**Liquid**	
$$\text{Re}\,sidue, mg/L = \frac{Total\ wt. - Wt. of\ dish\ or\ filter}{Vol. of\ sample, L}$$	mg/L	
TOX	**Liquid**	**Solid**
$$TOX, \mu g/L = (C_1 + C_2 - 2C_3) \times \frac{1000mL}{Vol. of\ sample}$$ C1 = instrument reading of 1 column C2 = instrument reading of 2 column C3 = instrument reading of blank column $$TOX, mg/kg = \frac{instrument\ reading}{\mu L\ injected} \times \frac{5}{dry\ wt.\ fraction}$$	mg/L	mg/Kg

12.5 Data Transfer and Review

12.5.1 Data Transfer to LIMS

The analytical results are entered on the department worksheets after review or by direct electronic transfer from the instrument data system. After the data are entered into the LIMS, they are checked against the information entered into the LIMS for transfer errors and anomalies.

12.5.2 Data Review

Laboratory analytical results are reviewed by a second analysts or a section supervisor. Prior to entering the reportable data into the LIMS, laboratory raw data have been reviewed, stamped, and signed to ensure that all of the method specifications have been met. This includes checking the extraction, digestion, distillation, and other preparation logs, as well as ensuring that all precision and accuracy requirements are addressed, and all steps of the analyses have been completed. If any problems arise during the analysis of the sample batch, it is the responsibility of the analyst and the section supervisor to bring this to the attention of the project manager, section manager, and QA manager through a written corrective action report.

The field/sampling manager is responsible for data review of all field-generated data. This includes verifying that all field descriptive data are recorded as per Section 6, that all field calibration requirements have been met as per Section 9, that all field QC data have met criteria given in Section 5, and that field data are entered accurately on worksheets.

Data flags are used on reports as needed to inform the project manager and the client of any additional information that might aid in the interpretation of the data. The data flagging system incorporates data qualifiers which are similar to flags specified in the Contract Laboratory Program protocols, as well as additional flags used to help explain batch specific events.

When data acquisition and reporting have been completed, the project manager reviews and prepares the final report. Because the project managers have extensive experience in evaluating analytical data, they have developed both objective and subjective techniques for data review. Each value reported is reviewed in the context of the respective environmental matrix and all available QC/QA data. Outliers or other abnormal values are carefully scrutinized, and samples are reanalyzed if the abnormalities cannot be explained. Where there are cases in which the results from spiked samples suggest interferences, attempts are made to remove the interferences, or alternate analytical procedures are used. If the interference problem cannot be resolved, the data are flagged and/or a narrative is included with the report.

12.5.3 Special Project or Data Package Review

If special handling and/or data packages are requested by the client, QA personnel also review the project report and the raw data. This includes checking that holding time requirements are met, checking calibrations, reviewing all quality control data and/or control charts, and initiating any corrective action or reanalyses that might be appropriate.

12.6 Reporting

The final report is printed and signed by the project manager after all review has been completed. The data flags that may appear in a project report are defined on the signature page, and any additional comments are also footnoted on this page.

If requested by the client or a project specific QA Plan, custom reports or CLP data packages with diskette deliverables can be provided. If data packaging is requested, a paginated data package is provided in addition to the project report. The format of the project report and/or data package can be adjusted to meet the needs of the client. All LIMS reports can be downloaded onto diskettes or to most clients' computers.

12.7 Data Storage

The procedures and policies for raw data retention are described in SL SOP QC16: *Analytical Records Maintained by SL* and summarized briefly as follows. After the projects are completed, the data are transferred to a secured area and filed chronologically by laboratory section in boxes and maintained for five years or the term specified in a client contract. In cases where data are reviewed on a computer screen, and a tape back-up system is available, electronic files of data are stored on tape in lieu of paper data for a period of ten years. If the data are to be purged to the client or need to be separated from the general raw data files, the data can be boxed, labeled and stored in a separate secured area. Keys to the data storage areas are retained by the QA staff and the section/department managers.

All in-lab data generated by computer systems are stored to tape or on hard disk, when the capability exists. The tapes are labeled and stored at the individual work stations or maintained by a data systems manager and serve as the lab's raw data files.

Hard copies of all LIMS reports are maintained for five years in client files. All LIMS reports and associated QC data are kept for a minimum of three years on the LIMS hard diskettes and/or magnetic tape. All data on the LIMS are backed up daily on magnetic tape.

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13.0 NONCONFORMANCE AND CORRECTIVE ACTION PROCEDURES

A nonconformance is defined as any occurrence that prevents the lab from delivering data that are compliant with the control criteria published or incorporated by reference in an applicable quality assurance plan. The non-conformance report (NCR) form (Figure 13.1) is used to document nonconformance conditions and to specify the necessary action(s) taken to correct the specific problem. The corrective action report (CAR) form (Figure 13.2) is used in situations where a recurring problem or breakdown in systems is observed and warrants a more thorough investigation than a single event NCR. CARs may be initiated from:

- a specified NCR
- an observed trend or frequency of events that warrant corrective action
- an audit finding

Savannah Laboratories will abide by all reasonable corrective actions generated from documented findings by agency audits

Some situations that develop and require formal documentation may not be appropriate for an NCR and CAR. Some of these anomalous situations are detailed on the Anomaly Report (Figure 13.3).

The status of all NCRs and CARs are tracked in registries located in various departments or centrally located in the lab. Summaries of the NCRs and CARs are provided to the Lab Director and management periodically so that overall trends in nonconformances and corrective actions can be evaluated. The procedures for preparation, tracking and disposition of NCRs, CARs, and Anomaly Reports are given in SL SOP CA85: *Nonconformance and Corrective Action Procedures.*

Table 13.1 summarizes the checks, the acceptance criteria, and the recommended corrective action for various QC activities. This table and SL SOP AN02: *Analytical Batching* are used to evaluate sample and batch QC.

FIGURE 13.1

Nonconformance Report (NCR)

Initiated by:__________ Date initiated:__________ Client:__________ NCR #:__________

Issuing department/(division): ________ Project manager/(division) :________ Method:______

Project/SDG#:__________ Sample(s) affected:______________ Batch ID:________

1	Nonconformance condition: Indicate and describe details (if necessary) Describe reason problem occurred (root cause):	☐Holding time ☐Catastrophic Failure ☐LCS/MS ☐Method blank ☐Calibration ☐Internal standards ☐Surrogates ☐Other
2	Action taken (add details if necessary): PM Initials/date: TM/DM/PS Initials/date:	☐Proceed with analysis ☐Reprep/reanalyze ☐Do not analyze ☐Case narrative (discuss in Sect. 5) ☐No action taken (justify)
3	Result of action taken in 2, above (add details if necessary):	☐Reprep/reanalysis acceptable ☐Reprep/reanalysis agrees w/ original ☐Other
4	Case Narrative Comments (if necessary) Initials/date:	
5	Close-out: Additional corrective action required to prevent recurrence? ☐YES ☐NO If YES, initiate corrective action report (CAR): CAR Initiated #: __________ Closed by: Date:	
6	**Original** to TM/DM; Copy to PM; Copy to RP (QC level III/IV only)	

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FIGURE 13.2

Corrective Action Report (CAR)

CAR #:_______________ Initiated by/date:_________________

Lab Director attn. requested? ☐Yes ☐No (*if yes, copy LD after completing section 1, below*)

1	Responsible TM/DM - summarize non-conformance incident and comments:
2	State root cause Initials: Date:
3	CORRECTIVE ACTION Assigned To:______________________________Target completion date:____________ Corrective action has been completed on (date):____________ By (initials):
4	QA Department Comments By (initials): Date:
5	Follow-up dates / comments (if no comments, indicate as "none") Initial 2 week 2 month ☐Add to internal systems checklist?

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FIGURE 13.3

ANOMALY REPORT

Date: ________ Log #: ________ Sample ID: ________ Client: ________

Dept: ____ EX ____ GE ____ LC ____ ME ____ RA ____ CU ____ SG ____ SM ____ VG ____ VM ____ AI

Analysis: ________

Reported by: ________

Anomaly:

☐Sample matrix is different than indicated by log-in.

Logged in as	*Best described as*
Water	*Water*
Soil	*Non-aqueous liquid*
Oil	*Soil*
	Sludge
	Oil
	Product
	Other ________

☐Sample was received with inadequate preservation, and was preserved upon receipt.

☐Sample received in an incompatible sample container. ____ *glass* ____ *plastic* ____ *other* ________

☐MS/MSD failed while the LCS/LCSD passed criteria, for a drinking water parameter. Method indicates data flagging.

☐Target analyte(s) detected in drinking water sample. (Describe below)

☐Sample exhibits gross non-homogeneity. (Describe below)

☐Insufficient sample received for analysis.

☐Data flag may be needed. Discuss with DM/LM before reporting.

☐Grand Mean exception was utilized for Initial Calibration (specify compounds).

☐Grand Mean exception was utilized for Continuing Calibration (specify compounds).

Other ________________________________

Custody: ****ALWAYS ATTACH A COPY OF COC WITH HIGHLIGHTED DEFICIENCY***

☐Sample description discrepancy between COC & Container	☐Custody seals broken
☐Sample container breakage	☐Incomplete COC
☐Cooler temp >6°C or frozen	☐Sample container partially filled
☐Sample received not listed on COC	☐Improperly preserved sample

Comments:

Client Notified: ☐Yes ☐No
Contact:

Date:

Resolution:

Route to:
Division PM:

Other Div. PM: SL________ ML________ NL________ FL________ TL________ BL________

STL

TABLE 13.1CORRECTIVE ACTION		
QC Activity	**Acceptance Criteria**	**Recommended Corrective Action**
GC/MS tuning	Section 9.0	Do not analyze samples unless criteria are met.
Initial calibration standards	Section 9.0	Reanalyze standards. If still unacceptable, remake standards or instrument corrections.
Continuing calibration standard	Section 9.0	Reanalyze standard. If still unacceptable, remake standards, or recalibrate.
Calibration blanks	< RL or QAPP/Method-defined criteria (for CLP procedures, use SOW guidelines)	Reanalyze calibration blank. If problem, determine source of contamination and reanalyze. Re-calibration may be required..
Method blank	< RL or QAPP/ Method-defined criteria (for CLP procedures, use SOW guidelines)	Reanalyze method blank. If problem, determine source of contamination. If necessary or possible, re-prep and re-analyze. Do not re-prep and re-analyze if no sample in batch or report contains the analyte(s) detected in the method blank. For SW-846 analyses, do not reanalyze if the method blank level is less than 5% of the regulatory limit or less than 5% of the lowest sample concentration .
Surrogate recovery (GC/MS semivolatiles)	Tables 5.1 - 5.8, or program/ project specific.	Follow method guidelines. Check calculations, check for possible matrix interferences, and if necessary or possible, extract sample and reanalyze
Surrogate recovery (GC/MS volatiles)	Tables 5.1 - 5.8, or program/project specific	Follow method guidelines. Check calculations, check for possible matrix interferences, and if possible, reanalyze sample
Surrogate recovery GC or LC	Tables 5.1 - 5.8, or program/project specific	Check for possible matrix interferences or other causes and follow method guidelines.
Matrix spike recoveries	Tables 5.1 - 5.8, or program/project specific	Check for possible matrix interferences or other causes. If still out, evaluate LCS.(1)
Lab control standard (LCS) recoveries	Tables 5.1 - 5.8, or program/project specific	Check calculations, reanalyze standards, and if necessary or possible, redigest or extract batch and reanalyze.(1)
Precision of MS/MSD or sample duplicate	Tables 5.1 - 5.8, or QAPP Specific (used for evaluation but not control unless specified by the method)	Check calculation. Check for possible matrix interference or other causes.
Internal standards (organics)	Method or protocol-required limits	Follow method or protocol guidelines.
Trip blanks	≤ RL	Check related method blank for contamination.
Field Blanks	≤ RL	Check related method blank for contamination.
Equipment blanks	≤ RL	Check related method blank for contamination.
Field duplicates	Follow project/program requirements	Follow project/program requirements
Microbiology + and - controls for media	Should be + and -, respectively	Reject medium.
Microbiology duplicates	RPD with established limits	Follow agency requirements.

(1) See Section 11.2.1 for guidance on full list spikes and unstable compounds

TABLE 13.1CORRECTIVE ACTION		
QC Activity	**Acceptance Criteria**	**Recommended Corrective Action**
Sample results	Calibration	If the calibration fails for a target and the corresponding target is not detected, the results may be reported as < RL if the RL standard is analyzed and detected.
	Spike criteria limits	If a limited list MS or LCS is high biased and no targets are detected above the RL; results are reported as < RL. When a full compound spike is utilized, and the MS or LCS result is high biased, and the corresponding target is not detected, the result for the corresponding target is reported as <RL, regardless of the other targets.
	Surrogate criteria limits	If surrogate recovery is high biased and no target is detected, the results are reported as <RL.
External Quality control check samples	Defined by the program or project.	Defined by the program or project.

14.0 PERFORMANCE SYSTEM AUDITS

14.1 Internal System Audits

14.1.1 Laboratory Audits

Annual laboratory audits are conducted by the division QA Manager or QA Officer. The scope and depth of the audit are determined according to the requirements of the division. The system audit includes, but is not limited to:

- evaluation of the procedures and items listed on the audit checklist
- review of compliance with the SL SOPs
- review of the compliance with this quality assurance plan
- review of the training records
- review of the nonconformance and anomaly reports and follow up on corrective actions from previous audits, external audits, or PE samples.

Some items may require more frequent auditing to determine if non-compliant procedures have been corrected. The internal audit may be performed quarterly for one or two sections with the goal of auditing of all systems once per year.

A report of the internal systems audit is prepared and submitted to the lab director and to the Corporate QA Manager by April 15.

14.1.2 Corporate Systems Audit

A systems audit of each division is conducted annually by the Corporate QA Manager to determine if the procedures implemented by the SL divisions are in compliance with this quality assurance plan and the standard operating procedures (SOPs). This is primarily accomplished by the review of the following:

- the annual systems audit performed by the division QA Manager or QA Officer
- the findings and responses to external audits
- the results of PE samples
- summaries of nonconformance and corrective action taken by the lab.

The annual systems audit is performed by the Corporate QA Manager by May 15. The Corporate QA Manager may request additional information or documentation of implementation. If necessary, the Corporate QA Manager will schedule an on-site evaluation of the division laboratories.

14.1.3 Field Audits

An audit of the field sampling procedures is performed annually. These systems audits are conducted by the QA Officer, an external auditor, or a regulatory agency. The audit includes all aspects of field sampling operations. Section 6.0 of this document defines the elements that serve as a basis for this audit.

14.2 External System Audits

Each laboratory may be certified by a number of state agencies, governmental agencies or private certification programs. Most of these programs require continuing on-site system audits of the laboratory. The laboratories submit to these on-sites as required by these certifying agencies and organizations and respond to any noted nonconformances with corrective actions.

Field system audits are performed periodically by various federal and state regulatory agencies. Field sampling and documentation procedures are examined to ensure sampling is performed according to the agency protocols.

14.3 Performance Audits

14.3.1 Internal Performance Audits

Internal performance audits or evaluations are routinely performed by Savannah Laboratories. Single blind performance audits are employed for several reasons. One purpose is to provide corrective action for parameters judged to be unacceptable on WP, WS or other major external performance audits. Periodic internal performance audits are also used to test parameters that are not routinely tested by external performance audits. Finally, single blind performance audits are employed to satisfy certain certification requirements, to satisfy auditors' specific requests for performance audit samples, or provide additional evidence of data quality to clients with specific questions regarding laboratory performance.

14.3.2 External Performance Audits

All facilities participate in each of the following performance evaluation audits semiannually:

1. U.S. EPA Water Supply Study (WS Series).
2. U.S. EPA Water Pollution Study (WP Series).

All facilities participate on an annual basis in a microbiology proficiency testing program.

Additionally, the laboratories participate in several regulatory agency, certifying group, or client requested performance audits. These performance audits include both single and double blind P.E. samples. Internal performance audits are logged into the LIMS system and analyzed and reported in the same manner as samples. Results from these performance audits are reported to management, agencies, and clients as required. Nonconformance Reports (NCR) and Corrective Action Reports (CAR) are issued when appropriate.

Results from agency performance audits are supplied to clients upon request.

15.0 QUALITY ASSURANCE REPORTS

15.1 Internal Reports

The QA Officer or QA Manager of each division is responsible for providing quality assurance reports to the division Lab Director and to the Corporate QA Manager on an annual basis. The report must include the following elements:

- complete internal audit checklist
- summaries of nonconformance/corrective actions from routine lab operations
- finding and responses to external audits and PE samples with any nonconformance/corrective action initiated
- results from all PE samples (internal and external)

These annual reports will be summarized by the Corporate QA Manager and provided to the President and Vice Presidents for review by May 15.

15.2 External Reports

Quality assurance or program reports are made to all required agencies or offices. The scope, content, and frequency of these reports are generally defined by the agency or office.

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16.0 TRAINING AND QUALIFICATIONS

STL-Savannah Laboratories' greatest asset is its well qualified and trained staff. The duties and responsibilities of management and staff positions are described in Section 4.0 of this manual. In addition, the qualifications for each of these positions can be obtained from the Corporate Human Resources Director at the Savannah Division.

STL-SL SOP CA01 (*SL Training SOP)* describes the procedures and documentation required to adequately train the analytical staff. All new employees are required to undergo an introduction to Savannah Labs' policies described in SL SOP CA10: *Procedures for New Employee Orientation*.

17.0 DOCUMENTS AND RECORDS

All documentation and records are maintained in accordance with SL SOPs AN45:*Laboratory Notebooks,*CA02:*Divisional Document Control and Distribution,* and CA80:*Data Generation, Entry, Review, Approval, and Reporting..*

Section 12 (Data Reduction, Review, and Reporting) of this manual describes the flow of data through the laboratory and the SL policy for document and record retention (Section 12.7). SL SOP QC16: *Analytical records Maintained by SL* describes the policies and procedures for the retention of analytical records (raw data and associated QC data).

18.0 PROCUREMENT

The SL policies and procedures for procurement are described in SL SOP CA 45: *Procurement.*

19.0 QUALITY IMPROVEMENT/MANAGEMENT ASSESSMENT

Savannah Laboratories and Environmental Services, Inc., is committed to quality improvement and customer service. All aspects of the laboratories operations are monitored and input from clients is evaluated to determine if present policies and procedures are meeting the objectives defined in Section 3.0 of this manual. The following SOPs have been implemented to address and document quality improvement:

Procurement of Laboratory Materials-SL SOP CA45: *Procurement*
Analytical Training- SL SOP CA01: *SL Training SOP*
Client Satisfaction and Complaint Resolution - SL SOP CA95: *Complaint Resolution*
Non-Conformance and Corrective Action - SL SOP CA85:*Noncomformance and Corrective Action Procedures*
Auditing (Divisional and Corporate) - SL SOP CA05:*Technical and Systems Audits (Divisional and Corporate)*

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Table of Target Analytes

Semivolatiles - GC/MS	Target Compound List[1] Method 8270 or CLP OLM03.2	Priority Pollutant List[2] Method 8270 or 625	Appendix IX List[3] Method 8270
Acenaphthene	X	X	X
Acenaphthylene	X	X	X
Acetophenone			X
2-Acetylaminofluorene			X
4-Aminobiphenyl			X
Aniline			X
Anthracene	X	X	X
Aramite (total)			X
Benzidine		X	
Benzo(a)anthracene	X	X	X
Benzo(b)fluoranthene	X	X	X
Benzo(k)fluoranthene	X	X	X
Benzo(g,h,i)perylene	X	X	X
Benzo(a)pyrene	X	X	X
Benzyl alcohol			X
4-Bromophenylphenyl ether	X	X	X
Butylbenzylphthalate	X	X	X
Carbazole	X		
4-Chloroaniline (p-Chloroaniline)	X		X
bis(2-Chloroethoxy)methane	X	X	X
bis(2-Chloroethyl)ether	X	X	X
4-Chloro-3-methylphenol (p-Chloro-m-cresol)	X	X	X
2-Chloronaphthalene	X	X	X
2-Chlorophenol	X	X	X
4-Chlorophenylphenyl ether	X	X	X
Chrysene	X	X	X
Diallate (total)			X
Dibenzo(a,h)anthracene	X	X	X
Dibenzofuran	X		X
Di-n-butylphthalate	X	X	X
1,2-Dichlorobenzene (o-Dichlorobenzene)	X	VOA**	X
1,3-Dichlorobenzene (m-Dichlorobenzene)	X	VOA**	X
1,4-Dichlorobenzene (p-Dichlorobenzene)	X	VOA**	X
3,3'-Dichlorobenzidine	X	X	X

Semivolatiles - GC/MS	Target Compound List[1] Method 8270 or CLP OLM03.2	Priority Pollutant List[2] Method 8270 or 625	Appendix IX List[3] Method 8270
2,4-Dichlorophenol	X	X	X
2,6-Dichlorophenol			X
Diethylphthalate	X	X	X
p-(Dimethylamino)azobenzene			X
7,12-Dimethylbenz(a)anthracene			X
3,3'-Dimethylbenzidine			X
alpha,alpha-Dimethylphenethylamine			X
2,4-Dimethylphenol	X	X	X
Dimethylphthalate	X	X	X
m-Dinitrobenzene			X
4,6-Dinitro-2-methylphenol (4,6-Dinitro-o-cresol)	X	X	X
2,4-Dinitrophenol	X	X	X
2,4-Dinitrotoluene	X	X	X
2,6-Dinitrotoluene	X	X	X
Dinoseb			X
1,4-Dioxane			X
1,2-Diphenylhydrazine		X	
Di-n-octylphthalate	X	X	X
bis(2-Ethylhexyl)phthalate	X	X	X
Ethyl methanesulfonate			X
Fluoranthene	X	X	X
Fluorene	X	X	X
Hexachlorobenzene	X	X	X
Hexachlorobutadiene	X	X	X
Hexachlorocyclopentadiene	X	X	X
Hexachloroethane	X	X	X
Hexachlorophene			X
Hexachloropropene			X
Indeno(1,2,3-cd)pyrene	X	X	X
Isophorone	X	X	X
Isosafrole			X
Methapyrilene			X
3-Methylcholanthrene			X

Semivolatiles - GC/MS	Target Compound List[1] Method 8270 or CLP OLM03.2	Priority Pollutant List[2] Method 8270 or 625	Appendix IX List[3] Method 8270
Methyl methanesulfonate			X
2-Methylnaphthalene	X		X
2-Methylphenol (o-Cresol)	X		X
3-Methylphenol (m-Cresol)			X
4-Methylphenol (p-Cresol)	X		X
Naphthalene	X	X	X
1,4-Naphthoquinone			X
1-Naphthylamine			X
2-Naphthylamine			X
2-Nitroaniline (o-Nitroaniline)	X		X
3-Nitroaniline (m-Nitroaniline)	X		X
4-Nitroaniline (p-Nitroaniline)	X		X
Nitrobenzene	X	X	X
2-Nitrophenol (o-Nitrophenol)	X	X	X
4-Nitrophenol (p-Nitrophenol)	X	X	X
5-Nitro-o-toluidine			X
4-Nitroquinoline-1-oxide			X
N-Nitrosodi-n-butylamine			X
N-Nitrosodiethylamine			X
N-Nitrosodimethylamine		X	X
N-Nitrosomethylethylamine			X
N-Nitrosodiphenylamine	X	X	X
N-Nitrosodi-n-propylamine	X	X	X
N-Nitrosomorpholine			X
N-Nitrosopiperidine			X
N-Nitrosopyrrolidine			X
2,2'-Oxybis(1-chloropropane)[bis(2-Chloroisopropyl)ether]	X	X	X
Pentachlorobenzene			X
Pentachloronitrobenzene			X
Pentachlorophenol	X	X	X
Phenacetin			X
Phenanthrene	X	X	X
Phenol	X	X	X
1,4-Phenylenediamine (p-Phenylenediamine)			X

Semivolatiles - GC/MS	Target Compound List[1] Method 8270 or CLP OLM03.2	Priority Pollutant List[2] Method 8270 or 625	Appendix IX List[3] Method 8270
2-Picoline			X
Pronamide			X
Pyrene	X	X	X
Pyridine			X
Safrole			X
1,2,4,5-Tetrachlorobenzene			X
2,3,4,6-Tetrachlorophenol			X
o-Toluidine			X
1,2,4-Trichlorobenzene	X	X	X
2,4,5-Trichlorophenol	X		X
2,4,6-Trichlorophenol	X	X	X
1,3,5-Trinitrobenzene			X
O,O,O-Triethyl phosphorothioate			X
Surrogates:			
2-Fluorobiphenyl	X	X	X
2-Fluorophenol	X	X	X
Nitrobenzene-d5	X	X	X
Phenol-d5	X	X	X
Terphenyl-d14	X	X	X
2,4,6-Tribromophenol	X	X	X
2-Chlorophenol-d4 (CLP only)	X (CLP)		
1,2-Dichlorobenzene-d4 (CLP only)	X(CLP)		

**VOA - For Priority Pollutants, Dichlorobenzenes are routinely analyzed as volatiles by EPA Method 8260 or EPA Method

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.2/ILM03.0/4.0

[2]Priority Pollutant List: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX List: 40 CFR 264, Appendix IX, 7/1/97

Table of Target Analytes

Chlorinated Dioxins and Furans	Priority Pollutant List[2] Method 613	Appendix IX List[3] Method 8280	SL Routine List Method 8280
Tetrachlorodibenzo-p-dioxins		X	X
Tetrachlorodibenzofurans		X	X
Pentachlorodibenzo-p-dioxins		X	X
Pentachlorodibenzofurans		X	X
Hexachlorodibenzo-p-dioxins		X	X
Hexachlorodibenzofurans		X	X
Heptachlorodibenzo-p-dioxins			X
Heptachlorodibenzofurans			X
Octachlorodibenzo-p-dioxins			X
Octachlorodibenzofurans			X
2,3,7,8-Tetrachlorodibenzo-p-dioxin	X	X	X

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.2/ILM03.0/4.0

[2]Priority Pollutant List: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX List: 40 CFR 264, Appendix IX, 7/1/97

Table of Target Analytes

Chlorinated Pesticides	Target Compound List[1] Method 8081 or CLP OLM03.2	Priority Pollutant List[2] Method 608	Appendix IX List[3] Method 8081
Aldrin	X	X	X
alpha-BHC	X	X	X
beta-BHC	X	X	X
gamma-BHC (Lindane)	X	X	X
delta-BHC	X	X	X
Chlordane (technical)		X	X
alpha-Chlordane	X		
gamma-Chlordane	X		
Chlorobenzilate			X
4,4'-DDD	X	X	X
4,4'-DDE	X	X	X
4,4'-DDT	X	X	X
Dieldrin	X	X	X
Endosulfan I	X	X	X
Endosulfan II	X	X	X
Endosulfan sulfate	X	X	X
Endrin	X	X	X
Endrin aldehyde	X	X	X
Endrin ketone	X		
Heptachlor	X	X	X
Heptachlor epoxide	X	X	X
Isodrin			X
Kepone			X
Methoxychlor	X		X
Toxaphene	X	X	X
Surrogates:			
Decachlorobiphenyl	X	X	X
Tetrachloro-m-xylene	X	X	X

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.2/ILM03.0/4.0

[2]Priority Pollutant List: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX List: 40 CFR 264, Appendix IX, 7/1/97

PCBs	Target Compound List[1] Method 8082 or CLP OLM03.2	Priority Pollutant List[2] Method 608	Appendix IX List[3] Method 8082
Aroclor-1016	X	X	X
Aroclor-1221	X	X	X
Aroclor-1232	X	X	X
Aroclor-1242	X	X	X
Aroclor-1248	X	X	X
Aroclor-1254	X	X	X
Aroclor-1260	X	X	X

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.2/ILM03.0/4.0

[2]Priority Pollutant List: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX List: 40 CFR 264, Appendix IX, 7/1/97

Chlorinated Herbicides	Appendix IX List[3] Method 8151	SL Routine List Method 8151
2,4-D	X	X
2,4-DB		X
2,4,5-T	X	X
2,4,5-TP (Silvex)	X	X
Dalapon		X
Dicamba		X
Dichloroprop		X
Dinoseb		X
MCPA [(4-chloro-2-methylphenoxy)- acetic acid]		X
MCPP [2- (4-chloro-2-methylphenoxy)- propanoic acid]		X
Pentachlorophenol		X
Surrogates:		
2,4-Dichlorophenyl acetic acid (DCAA)	X	X

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.2/ILM03.0/4.0

[2]Priority Pollutant List: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX List: 40 CFR 264, Appendix IX, 7/1/97

Organophosphorus Pesticides	Appendix IX List[3] Method 8141	SL Routine List Method 8141
Azinphos methyl		X
Bolstar (sulprofos)		X
Chlorpyrifos		X
Coumaphos		X
Demeton-O		X
Demeton-S		X
Diazinon		X
Dichlorvos		X
Dimethoate	X	X
Disulfoton	X	X
EPN		X
Ethoprop		X
Ethyl parathion (Parathion)	X	X
Famphur	X	X
Fensulfothion		X
Fenthion		X
Malathion		X
Merphos		X
Methyl parathion	X	X
Mevinphos		X
Monochrotophos		X
Naled		X
Phorate	X	X
Ronnel		X
Stirophos		X
Sulfotepp (Tetraethyl dithiopyrophosphate)	X	X
Thionazin (O,O-Diethyl-O-pyrazinyl phosphorothioate)	X	X
Tokuthion		X
Trichloronate		X
Surrogates:		
Triphenylphosphate	X	X

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.2/ILM03.0/4.0

[2]Priority Pollutant List: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX List: 40 CFR 264, Appendix IX, 7/1/97

Volatiles	Target Compound List[1] Method 8260 or CLP OLM03.2	Priority Pollutant List[2] Method 624 or 8260	Appendix IX List[3] Method 8260
Acetone	X		X
Acetonitrile			X
Acrolein (Propenal)		X	X
Acrylonitrile		X	X
Benzene	X	X	X
Bromodichloromethane	X	X	X
Bromoform	X	X	X
Bromomethane (Methyl bromide)	X	X	X
2-Butanone (MEK)	X		X
Carbon disulfide	X		X
Carbon tetrachloride	X	X	X
Chlorobenzene	X	X	X
Chloroethane	X	X	X
2-Chloroethylvinyl ether		X	
Chloroform	X	X	X
Chloromethane (Methyl chloride)	X	X	X
3-Chloro-1-propene (Allyl chloride)			X
Dibromochloromethane	X	X	X
1,2-Dibromo-3-chloropropane			X
1,2-Dibromoethane (EDB)			X
Dibromomethane (Methylene bromide)			X
1,2-Dichlorobenzene	SV**	X	SV**
1,3-Dichlorobenzene	SV**	X	SV**
1,4-Dichlorobenzene	SV**	X	SV**
trans-1,4-Dichloro-2-butene			X
Dichlorodifluoromethane			X
1,1-Dichloroethane	X	X	X
1,2-Dichloroethane	X	X	X
1,1-Dichloroethene	X	X	X
cis-1,2- Dichloroethene	(*)	(*)	(*)
trans-1-2-Dichloroethene	(*)	X	X
1,2-Dichloroethene(total)	X		
1,2-Dichloropropane	X	X	X
cis-1-3-Dichloropropene	X	X	X
trans-1-3-Dichloropropene	X	X	
Ethylbenzene	X	X	X
Ethyl methacrylate			X
2-Hexanone	X		X

Table of Target Analytes

Volatiles	Target Compound List[1] Method 8260 or CLP OLM03.2	Priority Pollutant List[2] Method 624 or 8260	Appendix IX List[3] Method 8260
Iodomethane (Methyl iodide)			X
Isobutanol (Isobutyl alcohol)			X
Methacrylonitrile			X
Methylene chloride (Dichloromethane)	X	X	X
Methyl methacrylate			X
4-Methyl-2-pentanone (MIBK)	X		X
Pentachloroethane			X
Propionitrile			X
Styrene	X		X
1,1,1,2-Tetrachloroethane			X
1,1,2,2-Tetrachloroethane	X	X	X
Tetrachloroethene	X	X	X
Toluene	X	X	X
1,1,1-Trichloroethane	X	X	X
1,1,2-Trichloroethane	X	X	X
Trichloroethene	X	X	X
Trichlorofluoromethane			X
1,2,3-Trichloropropane			X
Vinyl acetate			X
Vinyl chloride	X	X	X
o-Xylene	(*)	(*)	(*)
m and p-Xylene	(*)	(*)	(*)
Xylenes (Total)	X	(*)	X
Surrogates:			
p-Bromofluorobenzene (CLP)	X	X	X
Dibromofluoromethane	X	X	X
1,2-Dichlorobenzene-d4	X	X	X
1,2-Dichloroethane-d4 (CLP only)	X (CLP)		
Toluene-d8 (CLP)	X	X	X

(*) These isomers are not on the referenced list, but are routinely reported when EPA Method 8260 or EPA Method 624 is us

(**) SV-Dichlorobenzenes are routinely analyzed as semivolatiles employing EPA Method 8270 or EPA Method 625.

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.2/ILM03.0/4.0

[2]Priority Pollutant List: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX List: 40 CFR 264, Appendix IX, 7/1/97

Table of Target Analytes

Volatile-GC	SL Routine List Method 8021
Benzene	X
Bromodichloromethane	X
Bromoform	X
Bromomethane (Methyl bromide)	X
Carbon tetrachloride	X
Chlorobenzene	X
Chloroethane	X
2-Chloroethylvinyl ether	X
Chloroform	X
Chloromethane	X
Dibromochloromethane (Methylene bromide)	X
1,2-Dichlorobenzene	X
1,3-Dichlorobenzene	X
1,4-Dichlorobenzene	X
1,1-Dichloroethane	X
1,2-Dichloroethane	X
1,1-Dichloroethene	X
cis-1,2-Dichloroethene	(*)
trans-1,2-Dichloroethene	X
1,2-Dichloropropane	X
cis-1,3-Dichloropropene	X
trans-1,3-Dichloropropene	X
Ethylbenzene	X
Methylene Chloride (Dichloromethane)	X
Methyl tert-butyl ether (MTBE)	(*)
1,1,2,2-Tetrachloroethane	X
Tetrachloroethene	X
Toluene	X
1,1,1-Trichloroethane	X
1,1,2-Trichloroethane	X
Trichloroethene	X
Vinyl Chloride	X
Xylenes, total	(*)

(*) These compounds are not on the Priority Pollutant List, but are routinely reported when EPA Method 8021 is used.

Table of Target Analytes

Metals	Target Compound List[1] Methods 6010/7470/ 9012 or CLP ILM03.0/4.0	Priority Pollutant List[2] Methods 6010/ 7470/ 9012	Appendix IX List[3] Methods 6010/ 7470/ 9012
Aluminum	X		
Antimony	X	X	X
Arsenic	X	X	X
Barium	X		X
Beryllium	X	X	X
Cadmium	X	X	X
Calcium	X		
Chromium	X	X	X
Cobalt	X		X
Copper	X	X	X
Iron	X		
Lead	X	X	X
Magnesium	X		
Manganese	X		
Mercury	X	X	X
Nickel	X	X	X
Potassium	X		
Selenium	X	X	X
Silver	X	X	X
Sodium	X		
Thallium	X	X	X
Tin			X
Vanadium	X		X
Zinc	X	X	X
Cyanide	X	X	

Source of Lists:

[1]Target Compound List: CLP SOW OLM03.2/ILM03.0/4.0

[2]Priority Pollutant List: 40 CFR 423, Appendix A, 7/1/97

[3]Appendix IX List: 40 CFR 264, Appendix IX, 7/1/97

Metals	Regulatory Levels (mg/L)	Toxic Charcteristic Leaching Procedure (TCLP) Target Compounds [4]	
		Method 1311/6010	Method 1311/7470
Arsenic	5.0	X	
Barium	100.0	X	
Cadmium	1.0	X	
Chromium	5.0	X	
Lead	5.0	X	
Mercury (Cold Vapor)	0.2		X
Selenium	1.0	X	
Silver	5.0	X	

Chlorinated Pesticides Semivolatile-GC	Regulatory Levels (mg/L)	Toxic Charcteristic Leaching Procedure (TCLP) Target Compounds [4]
		Method 1311/8081
Endrin	0.02	X
Lindane	0.4-	X
Methoxychlor	10.0	X
Chlordane	0.03	X
Toxaphene	0.5	X
Heptachlor	0.008	X
Heptachlor Epoxide	0.008	X
Surrogates:		
Tetrachloro-m-xylene (TCMX)		X
Decachlorobiphenyl (DCB)		X

Source of List:

[4]TCLP Target Compound List: 40 CRF Part 261

Chlorinated Herbicides Semivolatile-GC	Regulatory Levels (mg/L)	Toxic Charcteristic Leaching Procedure (TCLP) Target Compounds [4]
		Method 1311/8151
2,4-D	10.0	X
2,4,5-TP (Silvex)	1.0	X
Surrogate:		
DCAA		X

Volatiles-GC/MS	Regulatory Levels (mg/L)	Toxic Charcteristic Leaching Procedure (TCLP) Target Compounds [4]
		Method 1311/8260
Benzene	0.5	X
Carbon tetrachloride	0.5	X
Chlorobenzene	100.0	X
1,2-Dichloroethane	0.5	X
Chloroform	6.0	X
1,1-Dichloroethylene	0.7	X
Methylethyl ketone	200.0	X
Trichloroethylene	0.5	X
Tetrachloroethylene	0.7	X
Vinyl chloride	0.2	X
Surrogates:		
Toluene-d8		X
p-Bromofluorobenzene		X
Dibromofluoromethane		X

Source of List:

[4]TCLP Target Compound List: 40 CRF Part 261

Semivolatiles-GC/MS	Regulatory Levels (mg/L)	Toxic Charcteristic Leaching Procedure (TCLP) Target Compounds [4] Method 1311/8270
Nitrobenzene	2.0	X
Hexachlorobenzene	0.13	X
1,4-Dichlorobenzene	2.5	X
2,4-Dinitrotoluene	0.13	X
Hexachlorobutadiene	0.5	X
Hexachloroethane	3.0	X
Pyridine	5.0	X
2,4,5-Trichlorophenol	400.0	X
2,4,6-Trichlorophenol	2.0	X
Cresols	200.0	X
Pentachlorophenol	100.0	X
Surrogates:		
Nitrobenzene-d5		X
2-Fluorobiphenyl		X
p-Terphenyl-d14		X
Phenol-d5		X
2-Fluorophenol		X
2,4,6-Tribromophenol		X

Source of List:

[4]TCLP Target Compound List: 40 CRF Part 261

ANALYTICAL SCHEME AND REPORT DELIVERABLE FORMAT FOR PCBs IN FISH AND OTHER BIOLOGICAL TISSUES

Approved by:

R. Wayne Robbins   8/19/99

Title: STL-SL QA Manager   Date

1.0 SCOPE AND APPLICATION

This document describes the general preparation and analytical procedures for the determination of polychlorinated biphenyls (PCB) in fish. Although the procedure is written specifically for the preparation and analysis of fish, other biological tissues may be prepared and analyzed in the same manner.

2.0 SUMMARY OF METHOD

Fish will be skinned or scaled as appropriate, filleted, packaged in aluminum foil and shipped on dry ice via express courier to STL-SL. One discrete fillet will be homogenized and analyzed by GC/ECD and/or SIM GC/MS utilizing this project specific document, PS15:10.11.96:1, which identifies the approach STL-SL will take with respect to PCB analysis in fish. The analytical approach is also based upon EPA methods 8081 (GC/ECD) and 680 (SIM-GC/MS). The remaining fillet will be archived and stored at < 0°C as a redundant sample in the event reanalysis of an unhomogenized fillet is required. A holding time of six months for the frozen fish fillets is recommended by Alabama Department of Environmental Management (ADEM) and will be used for all biological tissues.

3.0 PROCEDURE

3.1 Sample Preparation and Spiking Levels

The extraction of homogenized fish fillets will generally follow U. S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH, 1980 "Interim Methods for the Sampling Analysis of Priority Pollutants in Sediments and Fish Tissue" and the U.S. Environmental Protection Agency, Environmental Services Division, Region IV Method OB 10/90 (Attached) and Alabama Department of Environmental Management (ADEM) Preparation of Biological Tissue for Chemical Analysis (attached). Gel-permeation chromatography (GPC) will be employed as the primary cleanup technique utilized for the sample extracts. This cleanup procedure should eliminate most interferences; however, sulfuric acid cleanup will be employed.

A project specific reporting limit (RL) of 0.20ug/g for the individual polychlorinated biphenyl (PCB) mixtures (Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260) will be reported based on a hexane extraction of 10g of homogenized fish fillet tissue and analysis by GC/ECD. PCB analysis by SIM GC/MS is reported based on isomer groups (mono - decachlorobiphenyl). The reporting limit varies between the groups due to the varying levels of chlorination. Using the same extract as the GC/ECD procedure with further extract concentration for SIM GC/MS, the RLs for the various isomer groups are as follows:

PCB Isomer Group	RL For Each Isomer Group
monochlorobiphenyl - trichlorobiphenyl (Cl_1;Cl_2;Cl_3)	0.06 ug/g
tetrachlorobiphenyl - hexachlorobiphenyl (Cl_4;Cl_5;Cl_6)	0.12 ug/g
heptachlorobiphenyl - octachlorobiphenyl (Cl_7;Cl_8)	0.18 ug/g
nonachlorobiphenyl - decachlorobiphenyl (Cl_9;Cl_{10})	0.3 ug/g

The final extract volume (without accounting for GPC volume correction) will be 10.0mL for GC/ECD analyses and 1.0mL for SIM GC/MS analyses. An aliquot equal to roughly one-half of the 10.0mL final extract volume will be split out and further concentrated by a factor of ten for the SIM GC/MS analyses to yield the final volume equivalent concentration of 1.0mL, as previously specified.

GC V_f = 20mL accounting for GPC volume correction.

GC/MS V_f = 2.0mL accounting for GPC volume correction.

A percent lipid content will be evaluated on each discrete fillet as per the EPA procedure outlined in U.S. Environmental Protection Agency Environmental Services Division, Region IV Method OB 10/907.5 Determination of % Lipids (attached).

Sample aliquots will be weighed out in triplicate at the time of sample preparation so that two of the aliquots may be stored at < 0^0C in case they are needed for analysis at a later time.

PCB 1660 (a mixture of Aroclor 1016 and Aroclor 1260) will be used as the matrix spike/matrix spike duplicate and laboratory control spike. The spiked concentration in the fish will be 2.0ug/g resulting in an extract concentration of 1.0ug/mL for GC/ECD and 10ug/mL for GC/MS. This mixture encompasses the range of PCB congeners found within the list of Aroclors (GC/ECD) and PCB isomer groups (GC/MS) being analyzed. A homogenized fish fillet will be utilized for matrix QC purposes and an analyte-free aliquot of sodium sulfate will be prepared for the laboratory method blank and laboratory control spike. The recovery limits for the spikes are based upon the latest revision of the STL-SL LQM, Section 5.0, Table 5.5.

The isotope labeled surrogate $^{13}C_{12}$ Decachlorobiphenyl (DCB) will be added to all homogenized fish tissue aliquots and associated batch QC samples at a concentration of 0.20ug/g resulting in an extract concentration of 0.10ug/mL for GC/ECD and 1.0ug/mL for GC/MS. The recovery control limit of 30-150% for this surrogate is based on the CLP SOW Version OLM03.1 for the unlabeled DCB surrogate. Although the DCB surrogate recovery limit is advisory in the CLP SOW, this recovery limit will be required or appropriate corrective action (reanalysis or reextraction) will be taken.

3.2 Analytical Procedure

3.2.1 Gas Chromatography/Electron Capture (GC/EC)

The initial calibration (ICAL) will consist of a five point calibration for PCB1660 and a single mid-level calibration standard for the remaining Aroclor mixtures. A calibration check standard of PCB1660 will be analyzed every 20 samples or 12 hours, whichever is initially met. Additionally, the remaining 5 Aroclor mixtures will be analyzed prior to any samples which demonstrate the presence of these Aroclors in the sample extracts. The ICAL % relative standard deviation (RSD) criteria of ≤ 20% will be based on the mean %RSD of the calibration factors (CFs) generated from the 3-5 individual peaks evaluated for each level of the PCB1660 calibration. The mean %difference (%D) of the PCB1660 calibration check standard CFs must be ≤ 15%D from the ICAL average CFs. The single point ICAL for the other Aroclor mixtures is assumed valid when the PCB1660 calibration check standard meets criteria. If the calibration check standard fails criteria, the PCB1660 is recalibrated and the single level standard for the remaining Aroclors will also be reanalyzed (see GC/ECD analytical sequence on Page 3).

Quantitation of PCB1660 in the samples will be based on the average Cf from the PCB1660 ICAL. Quantitation of the remaining five Aroclor mixtures will be based on the single level CF which is generated from the analysis of these Aroclor mixtures in the same analytical clock as the sample extracts. The concentration of the upper calibration level (Level 5) for the PCB1660 will also be used as the extract concentration upper limit in determining when extract dilution may be required for any of the Aroclors. Up to five peaks will be evaluated for each Aroclor in the calibration standards and samples; and the average concentration from these peaks will be reported. When interference prohibits the use of any one peak, it will be eliminated when quantifying a specific Aroclor. In the presence of interferences, a minimum of three peaks may be evaluated when quantifying an Aroclor. If a minimum of three peaks without interference are not available for the identification and quantitation of individual Aroclor mixtures, then a SIM GC/MS procedure will be used for analysis and data reporting of the sample. In addition, SIM GC/MS confirmation will be performed on approximately 10% of the fish samples found to contain Aroclors at a level sufficient to be detected by GC/MS in the SIM mode. All samples analyzed and reported as greater than the RL by GC/ECD will be confirmed by GC/ECD on a second column regardless of whether the sample extract is analyzed by SIM GC/MS. Both the primary and confirmation results will be reported in addition to any SIM GC/MS which may also be available.

ANALYTICAL SEQUENCE

1) Instrument Blank
2) Aroclor 1221 mid-level calibration standard (1.0 ug/mL)
3) Aroclor 1232 mid-level calibration standard (1.0 ug/mL)
4) Aroclor 1242 mid-level calibration standard (1.0 ug/mL)
5) Aroclor 1248 mid-level calibration standard (1.0 ug/mL)
6) Aroclor 1254 mid-level calibration standard (1.0 ug/mL)
7) PCB1660 Level 1 (RL equivalent (0.2 ug/g) level calibration standard) (0.10 ug/mL)
8) PCB1660 Level 2 (0.25 ug/mL)
9) PCB1660 Level 3 (0.50 ug/mL)
10) PCB1660 Level 4 (calibration mid-level check standard concentration) (1.0 ug/mL)
11) PCB1660 Level 5 (2.5 ug/mL)
12) Instrument Blank
13) 20 field samples or 12 hours (whichever is initially met)
14) Additional Aroclor mixtures if so indicated
15) PCB1660 calibration check standard (Level 4)
16) Instrument Blank
17) 20 field samples or 12 hours (whichever criteria is initially met)
18) Follow steps 14 through 17 until recalibration is required, then reinitiate Step 1 of the sequence.

The following summary forms, associated standard, sample and QC chromatograms and an STL-SL Result Summary Report will be provided for the GC/ECD Level IV deliverable format:

1) **Form 1** PCB Aroclor Data Sheet
2) **Form 2** Surrogate Recovery
3) **Form 3** Matrix spike/Matrix spike duplicate for PCB 1660
   Lab Control Spike for PCB 1660
4) **Form 4** Method Blank Summary (Not to include samples analyzed & reported by GC/MS)
5) **Form 6** Initial Calibration (5 pt PCB1660 + 1 pt Aroclors 1221, 1232, 1242, 1248, 1254)
6) **Form 7** Calibration Check (1 point PCB1660)
7) **Form 8** Retention time shift (PCB analytical sequence) for $^{13}C_{12}$ DCB surrogate (Not to include samples analyzed & reported by GC/MS)
8) **Form 10** PCB ID Summary for multi-component analyses

3.2.2 Gas Chromatography/Mass Spectrometry using Selected Ion Monitoring (GC/MS-SIM)

The MS is hardware tuned and the tune is evaluated by the analysis of DFTPP in the GC/MS scan mode. The tune must meet the tuning criteria presented in method 680 in order to proceed with the analysis. Five acquisition groups are used to evaluate the quant, confirmation, and interference ions required to accurately identify and quantify any PCB congeners present. The start and stop times for the various acquisition groups are determined by running a calibration standard in scan mode and applying the procedure given in method 680 to generate the respective times. The scan standard is run on an as needed basis. The SIM start/stop times are verified before running client samples by a PCB window defining mix which contains the first and last eluting congener of each level of PCB chlorination.

After demonstrating that the MS is properly tuned and the acquisition method is switching between SIM groups at the correct times, an ICAL is analyzed. The ICAL consists of a five point calibration for each PCB isomer group using a single PCB congener from each group (excluding the nonachlorobiphenyl group which is calibrated based on the decachlorobiphenyl response). The mean response factor generated from the calibration for any one isomer group is used to quantify any PCB congener detected in that group. The ICAL %RSD response factor criteria is $\leq$ 30%. If this criteria is exceeded, additional analyses of the appropriate calibration levels may be analyzed to achieve an acceptable %RSD. Every 12 hours of sample analysis, a calibration check standard is analyzed and the RF for each isomer group must be within +/- 30% difference from the ICAL mean RF. If the calibration check standard fails criteria, a new ICAL is analyzed. Additional parameters are also evaluated for resolution between closely eluting congeners, signal to noise ratios (S/N), relative ion abundance criteria, and internal standard area in order to ensure that the GC/MS is operating properly.

ANALYTICAL SEQUENCE
1) DFTPP
2) Window Defining Mix
3) PCB Cal Level 1
4) PCB Cal Level 2
5) PCB Cal Level 3
6) PCB Cal Level 4
7) PCB Cal Level 5
8) Samples up to 12 hours from DFTPP
9) DFTPP
10) Window Defining Mix
11) PCB Calibration Check Standard (Cal Level 3)
12) Samples up to 12 hours from DFTPP
13) Follow steps 9 through 12 until recalibration is required, then reinitiate Step 1 of the sequence.

The following summary forms, associated standard, sample and QC chromatograms and an SL Result Summary Report will be provided for the SIM GC/MS Level IV deliverable format:

1) **Form 1** PCB Isomer Group Data Sheet
2) **Form 2** Surrogate Recovery - $^{13}C_{12}$ DCB
3) **Form 3** Matrix spike/Matrix spike duplicate for PCB 1660
   Lab Control Spike for PCB 1660
4) **Form 4** Method Blank Summary
5) **Form 5** DFTPP Instrument Performance Check
6) **Form 6** Initial Calibration (5 point - single PCB congeners for Cl_1 through Cl_8 and Cl_{10} PCB isomer groups)
7) **Form 7** Calibration Check (Cal Level 3)
8) **Form 8** Internal Standard Area Summary

Alternately an SL Level II (LIMS Summary only) or SL Level III format (LIMS Summary and Forms only) can be provided.

3.3 Method Detection Limit and Method Performance Studies

In accordance with 40 CFR Part 136, Appendix B, an MDL study will be performed for the PCB in fish tissue procedure by GC/ECD and GC/MS. This study will be based on data generated from 10 replicate spikes of the target analytes to be reported. This will involve an MDL for each specific Aroclor by GC/ECD and for each PCB isomer group by SIM GC/MS. Outliers will be evaluated using the statistically correct *Q-test*, as described by Dean and Dixon, *Anal. Chem.*, 23, 636 (1951). A minimum of 7 replicates must be statistically retained for the study to be valid as per Appendix B.

A method performance study involving the analysis of 4 replicate spikes of the target analytes at a mid-level concentration in the fish will be performed. This study will provide information on the precision and accuracy of this procedure with respect to fish tissue.

Summary Table of Spike Levels

Target Analyte	Study Type	Volume of spike added (mL)	Con. of spike soln. (ug/mL)	Spiked conc. in Fish (ug/g)	GC/ECD extract conc. (ug/mL)	GC/MS extract conc. (ug/mL)
$^{13}C_{12}$DCB	Sample/QC	1.0	2.0	0.20	0.10	1.0
PCB1660	MS/LCS	1.0	20	2.0	1.0	10
$^{13}C_{12}$DCB	MDL-GC	1.0	1.0	0.20	0.1	NA
Aroclor 1221	MDL-GC	1.0	1.0	0.20	0.1	NA
Aroclor 1232	MDL-GC	1.0	1.0	0.20	0.1	NA
Aroclor 1242	MDL-GC	1.0	1.0	0.20	0.1	NA
Aroclor 1248	MDL-GC	1.0	1.0	0.20	0.1	NA
Aroclor 1254	MDL-GC	1.0	1.0	0.20	0.1	NA
Aroclor 1660	MDL-GC	1.0	1.0	0.20	0.1	NA
$^{13}C_{12}$DCB	MPS-GC	1.0	1.0	0.20	0.1	NA
Aroclor 1221	MPS-GC	1.0	10	2.0	1.0	NA
Aroclor 1232	MPS-GC	1.0	10	2.0	1.0	NA
Aroclor 1242	MPS-GC	1.0	10	2.0	1.0	NA
Aroclor 1248	MPS-GC	1.0	10	2.0	1.0	NA
Aroclor 1254	MPS-GC	1.0	10	2.0	1.0	NA
Aroclor 1660	MPS-GC	1.0	10	2.0	1.0	NA
$^{13}C_{12}$DCB	MDL-GC/MS	1.0	1.0	0.20	NA	1.0
Cl_1-Cl_3	MDL-GC/MS	1.0	0.1	0.020	NA	0.1
Cl_4-Cl_6	COMBINED	|	0.2	0.040	NA	0.2
Cl_7-Cl_8	|	|	0.3	0.060	NA	0.3
Cl_{10}	|	|	0.5	0.10	NA	0.5
$^{13}C_{12}$DCB	MPS-GC/MS	1.0	1.0	0.20	NA	1.0
Cl_1-Cl_3	MPS-GC/MS	1.0	1.0	0.20	NA	1.0
Cl_4-Cl_6	COMBINED	|	2.0	0.40	NA	2.0
Cl_7-Cl_8	|	|	3.0	0.60	NA	3.0
Cl_{10}	|	|	5.0	1.0	NA	5.0

PCB 1660 (Aroclor 1016 & Aroclor 1260)

Approval Signature: R. Wayne Robbins	
Title: Corporate QA Manager	Date: July 15, 1999

Polychlorinated Biphenyls (PCBs) by GC/MS (Method 680)

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of polychlorinated biphenyls (PCBs) in groundwater, soils, sediments, wastes, and biological tissues by GC/MS. PCBs are reported by the level of chlorination: monochlorobiphenyls, dichlorobiphenyls, trichlorobiphenyls, etc., up to decachlorobiphenyl.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for each target compound is given in Section 5 of the current revisions of the Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is spiked with a surrogate and extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS operated in the Selected Ion Monitoring Mode (SIM). Windows are established to monitor for the characteristic masses of the various PCB congeners. Qualitative identification of the target compounds in the extract is based on the presence of the peak within the SIM window and the mass ratio between the primary and confirmation ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion. Results are reported as total monochlorobiphenyls, total dichlorobiphenyls, etc.

The default identification and quantitation procedure will be to use only the quantitation and confirmation ions for the PCB congeners. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB congeners unless specified in a client QAP or agency requirement or the sample concentration is near a critical quantitation limit. Samples evaluated according to the default quantitation procedures may be slightly high biased.

2.2 This procedure is based on the guidance provided in EPA Method 680. The method has been modified to include the use of a carbon-13 labeled analogue of decachlorobiphenyl (13C12-DCB) as the surrogate in place of the labeled BHC and DDT compounds. 13C12-DCB can be subjected to the optional acid cleanup (the unlabeled analogue, decachlorobiphenyl, is routinely used in SW-846 Method 8082). A window-defining mix containing the first and last eluting isomers of each level of chlorination is used as an aid to establish and verify that the SIM windows are properly set. Preparation procedures are also referenced for soils and biological tissues, which are not included in EPA Method 680.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.2 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products such as diesel or waste oil. GPC cleanup may be necessary for biological tissues.
Acid cleanup may be employed as an additional cleanup tool.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ Storage	Container	Sample Hold Time[1]	Extract Hold Time[1]
Aqueous	none; 4C +/- 2C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C +/- 2C	500-mL	14 days	40 days
Biological	Frozen	Glass or wrapped in aluminum foil	6 months	40 days

[1] Holding times are advisory - no holding times are defined in method 680.

6.0 APPARATUS AND MATERIALS

6.1 GC/MS System with compatible data system autosampler, splitless injector, and direct capillary interface.

6.2 Recommended Capillary column-HP-5MS, 30m x 0.25mm ID, 0.50um film thickness. Equivalent columns can be used.

6.3 Microsyringes-

6.4 Volumetric flasks, Class A-appropriate volumes

6.5 Analytical balance

7.0 REAGENTS

7.1 Hexane

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in SL SOP AN43:*Standard Preparation.*

The lab should purchase certified solutions from SL-approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SL SOP AN43 for guidance for standard preparation from neat materials.

8.1 The recommended calibration standards are listed in Appendix A, Table 1. Prepare these standards at the stated concentrations in hexane.

8.2 The surrogate compound, 13C12-Decachlorobiphenyl, is prepared at a concentration of 1.0ug/mL and 1.0mL of this solution is spiked into all samples and QC items prior to extraction.

8.3 The matrix spiking solution contains one PCB congener of each chlorination level except for the nonachlorobiphenyls. The solution is prepared at the indicated concentrations in acetone and 1.0mL of this solution is spiked into all lab spikes and matrix spikes.

COMPOUND	CONCENTRATION (ug/mL)
2-Chlorobiphenyl	2.0
2,3-Dichlorobiphenyl	2.0
2,4,5-Trichlorobiphenyl	2.0
2,2',4,6-Tetrachlorobiphenyl	4.0
2,2',3,4,5'-Pentachlorobiphenyl	4.0
2,2',4,4',5,6'-Hexachlorobiphenyl	4.0
2,2',3,4',5,6,6'-Heptachlorobiphenyl	6.0
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	6.0
Decachlorobiphenyl	10

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP Number	Extraction Technique
Aqueous	EX30	Continuous Liquid-liquid Extraction
Aqueous	EX35	Separatory Funnel
Soils/Sediments and Biological	EX40, PS15	Sonication

9.2 The sample concentration procedures are given in SL SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (SL SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products. Acid cleanup (SL SOP EX60) is recommended as a routine cleanup prior to analysis. Sulfur cleanup may be necessary if the sample extract contains high levels of sulfur.

10.0 PROCEDURE

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:
HP-5MS 30m x 0.25mm ID, 0.50um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:
Initial column temperature: 45 C for 1 minutes
Column temperature program 1 : 20C per minute to 150C, hold 1 minute
Column temperature program 2 : 10C per minute to 310C, hold until DCB and 13C12-DCB elute

GC injector parameters
Injector temperature : 250-260°C
Injector : splitless
Inlet purge time : 0.8 minutes
Injector liner : 4mm ID quartz or 4mm glass, deactivated
Sample injection volume : 2uL

Mass Spectrometer and interface parameters
Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: SIM (see Table 3 in Appendix B for ions to monitor)
Mass range for DFTPP analysis: 35-500amu at 1 scan per second or less.

10.2 Tune Criteria

Twenty nanograms of DFTPP are analyzed at the beginning of each 12 hour clock as a check on the "tune" of the mass spectrometer. The DFTPP analysis is performed using scan analysis and the same tune parameters are used for the SIM analysis of the calibration standards and samples.

10.2.1 Prepare a 10ng/uL solution of DFTPP column evaluation standard. The standard must also contain p,p'-DDT (4,4'-DDT).

10.2.2 Analyze a 1uL aliquot of the 10ng/uL DFTPP solution using the same temperature program that is used for SIM analysis of the calibration standards, samples, and QC samples.

10.2.3 Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.3 Window-Defining Solution and SIM Parameters

10.3.1 Analyze 1uL of the window-defining solutions in the scan mode from 45amu to 500amu at > 1 scan per second. Use the same temperature program that will be used for the SIM analysis of PCBs. The window defining solutions may be analyzed separately or may be combined into a single solution.

10.3.2 Determine the retention times of the first and last eluting congeners at each level of chlorination. The quantiation and confirmation masses are listed in Table 3 of Appendix B.

10.3.3 Set the SIM parameters as follows. Refer to Table 3 of Appendix B for the ion sets.

-Begin data acquisition with ion set #1 before the elution of PCB congener #1, Stop the acquisition of ion set #1 and begin acquisition of ion set #2 approximately 10 seconds before the elution of PCB congener #104.
-Stop the acquisition of ion set #2 and begin the acquisition of ion set #3 approximately 10 seconds after the elution of PCB congener #77..
-Stop the acquisition of ion set #3 and begin the acquisition of ion set #4 approximately 10 seconds after the elution of 4,4'-DDT. (the retention time of 4,4'-DDT is determined from the scan analysis of the DFTPP solution that is analyzed at the beginning of each 12-hour clock.).
-Stop the acquisition of ion set #4 and begin the acquisition of ion set #5 approximately 10 seconds before the elution of PCB congener #208.

10.4 Initial Calibration
After the SIM windows are established and verified and the DFTPP criteria has been met, the initial calibration standards are analyzed. Note that a single PCB congener of each chlorination level is used for calibration and quantiation. Decachlorobiphenyl is used to quantify nonachlorobiphenyls.

10.4.1 Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range.

10.4.2 Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts. The routine volume is 1uL.

10.4.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.4.4The relative response factor for each compound is calculated as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion of the calibration congener
Ais = area of the characteristic ion for Chrysene-d10
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

NOTE: Use Chrysene-d10 as the internal standard unless matrix interferences are encountered. If phenthrene-d10 must be used, the calibration must be re-evaluated and verified using the second internal standard

10.4.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3 .... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

10.4.6Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_l} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.4.7Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\, 100$$

- If the %RSD of each target compound is less than or equal to 20%, the average response factor can be used for quantitation of samples.

- If the %RSD of the target compound is greater than 20%, the curve should be evaluated for errors and one or more standards re-analyzed. Take corrective action until the %RSD of each target is less than 20%.

10.4.8 Performance Criteria
In addition to meeting the calibration criteria, the following performance criteria must also be met for the mid-level standard.

-Mass abundance ratios of all calibration congeners within acceptance range
(see Table 4,Appendix B)
-Baseline separation of PCB congener #87 from congeners #154 and #77
-Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241
-decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498

10.5 Continuing Calibration Verification
Samples are analyzed only after the DFTPP criteria and the calibration acceptance criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the DFTPP and a mid-level calibration standard prior tot he analysis of samples and after the samples by the analysis and evaluation of a mid-level standard.

10.5.1 The percent difference or percent drift between the continuing calibration RRF and the average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$\%difference = \left| \frac{RRF - RRFavg}{RRFavg} \right| \otimes 100$$

where
RRF = relative response factor from CCV
RRFavg = average relative response factor from initial calibration curve

If the percent difference is less than or equal to 20% for each target compound, the initial calibration is verified.

If the continuing calibration criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the analysis of the continuing calibration standard must be repeated. The analyst must be aware of the 12-hour clock-DFTPP criteria must be met prior to the analysis of the calibration standards. If the continuing calibration standard repeatedly fails the calibration verification criteria, the initial calibration curve must be reanalyzed and reevaluated.

The performance criteria given in Section 10.4.8 must also be met prior to the analysis of samples.

10.6 Sample Analysis
Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.6.1 Add 30uL of the internal standard mix (25ug/mL) to each 1-mL aliquot of the sample extract. The concentration of the internal standard in the extract is 0.75 ng/uL.

10.6.2 Mix the contents of the autosampler vial by inverting several times.

10.6.3 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

10.6.4 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract is diluted to final volume of 1.0mL, the dilution factor is 10. (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (25ug/mL)-Vistd	DF
1000	0	1000	30	1
500	500	1000	15*	2
200	800	1000	24	5
100	900	1000	27	10
50	950	1000	28.5	20
20	980	1000	30	50

*assumes dilution of a 1.0mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 0.75ug/mL using 30ul of a 25.0ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 0.75ug/mL. The following equation can be used to determine the volume of the 25.0 ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd(uL) = 30uL - \left(\frac{Vext}{Vdil} \otimes 30ul\right)$$

Vistd = volume of 25.0ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

The default procedure will be to use only the quantitation and confirmation ions for identification and quantification of PCB congeners. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB congeners unless specified in a client QAP or agency requirement or the sample concentration is near a critical quantitation limit.

11.1.1 Examine the Selected Ion Current Profiles (SICP) for the internal standards. Confirm that the RT and response of the internal standards are within the acceptance criteria specified in the SOP Summary. If the internal standard retention times have changed significantly or the peaks cannot be located, stop and analysis and correct the problem. Reanalyze any associated samples.

11.1.2 Evaluate the peaks for candidates to be identified as PCBs. A peak is tentatively identified as a PCB if

-The peak falls within the retention time range bordered by the first and last eluting isomer of that chlorination level

-The ratio of the quantitation and confirmation ions are present and the area ratios fall within the acceptance criteria in Appendix B, Table 4. The scans must maximize within one scan of each other. Examine the data for the presence of a coeluting PCB of higher chlorination if both ions and the M-70 ions are present and the ratio does fall within the acceptance limits.

-The areas for the quantiation and confirmation ions must be greater than three times the background noise and must fall within the working range of the calibration curve (must not saturate the detector

-At least one ion in the M-70 cluster must be present

11.1.3 Evaluate each PCB candidate in the Cl-3 to Cl-7 range for the presence of coeluting PCBs containing one or two additional chlorines. An intense M+35 ion at the retention time may indicate a PCB with one additional chlorine and the presence of an intense M+70 would indicate a co-eluting PCB containing two additional chlorines. Use the information in Tables 5 and 6 of Appendix B to correct for the interfering ion(s).

For example, if a Cl-7-PCB and a Cl-5-PCB coelute, the Cl-7-PCB will contribute to the quantiation and confirmation ions for the Cl-5-PCB. Cl-7-PCB produces a cluster of three ions by the loss of two chlorines-ions 322,324, and 326. Two of these ions-324 and 326-are also ions contained in the molecular ion cluster of Cl-5-PCB. To determine the ion 326 and 324 areas produced only by the Cl-5-PCB, calculate the contribution to each and subtract it from the measured areas. See Tables 5 and 6 in Appendix B for the percentage of the interference peak to subtract from the quantiation and confirmation ions. In this example, 164% of the area measured for ion 322 should be subtracted from the area measured for ion 324 and 108% of the area measured for ion 322 should be subtracted from ion 326.

NOTE: A coeluting PCB with one more chlorine will affect only the quantiation ion (Table 6).
The interference from a coeluting PCB containing one more chlorine, due to the natural abundance of 13C12, is small and can usually be neglected except when measuring the area of a small amount of a PCB coeluting with a large amount of a another PCB containing one more chlorine.

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples.

11.2.1 Aqueous Samples

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where

Ax =	sum of areas of the characteristic ion of the PCB chlorination level being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where

F = final volume of extract (mL)
Fqap = 1.0mL
Vqap = 1.0L
V = volume of sample extracted
DF = dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

$$concentration(ug/kg, dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	sum of areas of the characteristic ion of the PCB chlorination level being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100

The SL CQAP assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. SL SOP AN02 also describes the procedure for evaluating batch-specific QC. The attached SOP summary and Table 13.1 in the SL Corporate QA Plan provide guidance for evaluating sample data.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each laboratory must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration are the preparation and analysis of spiked reagent water. The criteria for IDOC accuracy and precision are the accuracy and precision criteria listed in Table 5 of the SL QAP.

12.3 Method Detection Limit

The method detection limit is determined by each lab annually in accordance with SL SOP CA90:*Procedure for Determination of Method Detection Limit (MDL)*.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. See Section 10 of the current SL Quality Assurance Plan.

14.0 TROUBLE-SHOOTING-Trouble-shooting items will be added at a later time.

15.0 REFERENCES

15.1 *Savannah Laboratories' Comprehensive Quality Assurance Plan* and Savannah Laboratories' *Corporate Quality Assurance Plan*, current revisions.

15.2 *Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry*. November 1985. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH

APPENDIX A

TABLE 1 CALIBRATION STANDARDS

CALIBRATION COMPONENTS	CAL 1	CAL 2	CAL3	CAL4	CAL5
Calibration Congener					
2-chlorobiphenyl (1)	0.10	0.50	1.0	2.0	5.0
2,3-dichlorobiphenyl(5)	0.10	0.50	1.0	2.0	5.0
2,4,5-trichlorobiphenyl(29)	0.10	0.50	1.0	2.0	5.0
2,2',4,6-tetrachlorobiphenyl(50)	0.20	1.0	2.0	4.0	10
2,2',3,4,5'-Pentachlorobiphenyl (87)	0.20	1.0	2.0	4.0	10
2,2',4,4',5,6'-hexachlorobiphenyl(154)	0.20	1.0	2.0	4.0	10
2,2',3,4',5,6,6'-heptachlorobiphenyl(188)	0.30	1.5	3.0	6.0	15
2,2',3,3',4,5,5',6,6'-octachlorobiphenyl(200)	0.30	1.5	3.0	6.0	15
Decachlorobiphenyl	0.50	2.5	5.0	10	25
Retention Time Congeners					
3,3',4,4-tetrachlorobiphenyl(77)	0.20	1.0	2.0	4.0	10
2,2',4,6,6'-Pentachlorobiphenyl (104)	0.20	1.0	2.0	4.0	10
2,2',3,3',4,5,5',6,7'-nonachlorobiphenyl(208)	0.40	2.0	4.0	8.0	20
Surrogate					
13C12-Decachlorobiphenyl	0.50	2.5	5.0	10	25
Internal Standards					
Phenathrene-d10	0.75	0.75	0.75	0.75	0.75
Chrysene-d12	0.75	0.75	0.75	0.75	0.75

TABLE 2 –First and Last Eluting Isomers

Congener	First Eluting Isomer	Last Eluting Isomer
Cl-1	2-chlorobiphenyl	4-Chlorobiphenyl
Cl-2	2,6-dichlorobiphenyl	4,4'-dichlorobiphenyl
Cl-3	2,2',6-trichlorobiphenyl	3,4,4'-trichlorobiphenyl
Cl-4	2,2',6,6'-tetrachlorobiphenyl	3,3',4,4'-tetrachlorobiphenyl
Cl-5	2,2',4,6,6'-pentachlorobiphenyl	3,3',4,4',5-pentachlorobiphenyl
Cl-6	2,2',4,4',6,6'-hexachlorobiphenyl	3,3',4,4',5,5'-hexachlorobiphenyl
Cl-7	2,2',3,4',5,6,6'-heptachlorobiphenyl	2,3,3',4,4',5,5'-heptachlorobiphenyl
Cl-8	2,2',3,3',5,5',6,6'-octachlorobiphenyl	2,3,3',4,4',5,5',6-octachlorobiphenyl
Cl-9	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl

APPENDIX B
SIM IONS

Table 3-Ions for SIM Acquisition

ION Set 1 (a)	ION Set 2 (b)	ION Set 3 (c)	ION Set 4 (d)	ION Set5 (e)
152	186	248	240	356
153	188	249	241	358
186	220	254	288	360
187	222	256	290	390
188	254	288	322	392
189	255	290	324	394
190	256	322	326	424
220	258	323	356	425
221	288	324	357	426
222	289	326	358	428
224	290	328	360	430
255	292	357	362	432
256	294	358	391	462
258	323	360	392	464
290	324	362	394	466
292	326	392	396	496
294	328	394	398	498
	358	396	428	499
	360	398	430	500
	362		432	502

(a) Cl-1 to Cl-4 and Phenthrene-d10
(b) Cl-3 to-Cl-6
(c) Cl5 to Cl-7
(d) Cl-6 to Cl-8 and Chrysene-d12
(e) Cl-8 to Cl-10 and 13C12-DCB

TABLE 4-Approximate Retention Times for PCB Isomer Groups and Calibration Congeners

PCB Isomer Group	Approximate RRT Range	Calibration Congener	Approximate Calibration Congener RRT
Cl-1	0.30-0.35	2-chlorobiphenyl (1)	0.30
Cl-2	0.38-0.50	2,3-dichlorobiphenyl(5)	0.43
Cl-3	0.46-0.64	2,4,5-trichlorobiphenyl(29)	0.54
Cl-4	0.55-0.82	2,2',4,6-tetrachlorobiphenyl(50)	0.56
Cl-5	0.64-0.92	2,2',3,4,5'-entachlorobiphenyl (87)	0.80
Cl-6	0.75-1.1	2,2',4,4',5,6'-hexachlorobiphenyl(154)	0.82
Cl-7	0.88-1.2	2,2',3,4',5,6,6'-heptachlorobiphenyl(188)	0.88
Cl-8	0.99-1.21	2,2',3,3',4,5,5',6,6'-octachlorobiphenyl(200)	1.03
Cl-9	0.16-1.28	Decachlorobiphenyl	1.3
Cl-10	1.3	Decachlorobiphenyl	1.3

RRT = retention time relative to Chrysene-d12

Table 4 Quantitation and Interference Check Ions

PCB Isomer Group	Quant ION	Confirmation ION	Expected Ratio(a)	Acceptable Ratio(a)	M-70 Confirmation ION	Interference Check ION M+70	Interference Check ION M+35
Cl-1	188	190	3.0	2.5-3.5	152	256	222
Cl-2	222	224	1.5	1.3-1.7	152	292	256
Cl-3	256	258	1.0	0.8-1.2	186	326	290
Cl-4	292	290	1.3	1.1-1.5	220	360	326
Cl-5	326	324	1.6	1.4-1.8	354	394	360
Cl-6	360	362	1.2	1.0-1.4	288	430	394
Cl-7	394	396	1.0	0.8-1.2	322	464	430
Cl-8	430	428	1.1	0.9-1.3	356	498	464
Cl-9	464	466	1.3	1.1-1.5	390		498
Cl-10	498	500	1.1	0.9-1.3	424		
Chrysene-d12	240	241	5.1	4.3-5.9			
Phenathrene-d10	188	189	6.6	6.0-7.2			
13C12-DCB (surrogate)	510	512					

(a) ratio of quantitation ion to confirmation ion

TABLE 5-Corrections for Interference of PCB Containing Two Additional Chlorines

PCB Isomer Group	Quant ION	Confirmation ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area	Percent Ion area to be subtracted from CONFIRMATION ION Area
Cl-3	256	258	254	99	33
Cl-4	292	290	288	65	131
Cl-5	326	324	322	108	164
Cl-6	360	362	356	161	71
Cl-7	394	396	390	225	123

TABLE 6-Corrections for Interference of PCB Containing One Additional Chlorine

PCB Isomer Group	Quant ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area
Cl-2	222	221	13.5
Cl-3	256	255	13.5
Cl-4	292	289	17.4
Cl-5	326	323	22.0
Cl-6	360	357	26.5
Cl-7	394	391	30.9
Cl-8	430	425	40.0

APPENDIX C
680 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Biological	Frozen	Glass or aluminum foil	6 months	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
DFTPP 10ng on column Clock starts at injection	DFTPP 10ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification
Samples and the capping standard must be analyzed within 12 hours of the start of the clock	Samples and the capping standard must be analyzed within 12 hours of the start of the clock
Capping standard	Capping standard

DFTPP CRITERIA

m/z	Ion Abundance Criteria
127	40-60% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass443
442	>40% of mass 198
443	17-23% of mass 442

ANALYTICAL BATCH
SEE SL SOP AN02.

CALIBRATION ACCEPTANCE CRITERIA

Initial Calibration	Continuing Calibration
RRFavg <= 20% RSD	Percent difference <= 20% difference from initial calibration

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 20ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
Initial Calibration-minimum of five calibration standards	After Tune Check and when calibration verification standard fails acceptance criteria.	%RSD <= 20%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Performance Criteria	Evaluate mid level calibration standard each clock	-Mass abundance ratios of all calibration congeners within acceptance range (see Appendix B) -Baseline separation of PCB congener #87 from congeners #154 and #77 -Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241 -decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples and at the end of the analytical sequence	%Difference <= 20%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

QC Item	Frequency	Acceptance Criteria	Corrective Action
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be within 30% of the previous CCV and within 50% of the initial calibration Areas in samples should be evaluated for gross error . Consult superior	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within Section 5 QAP limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1
Lab Control Standard (LCS) - QAP subset	See AN02	All spiked targets within the accuracy criteria in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See AN02	All targets within the accuracy and precision criteria in Section 5 Table of QAP	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SL SOP AN02 and SL QAP Table 13.1

QC Item	Frequency	Acceptance Criteria	Corrective Action
Initial Demonstration of Capability (IDOC)	Per analyst	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	See CA90	Evaluate according to SL SOP CA90	Evaluate according to SL SOP CA90

VOLATILE COMPOUNDS BY GC/MS (EPA 8260B)

Approved by:

R. Wayne Robbins — Nov 12, 1999

Title: STL-SL QA Manager — Date

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures that can be used to determine the concentration of volatile organic compounds (VOC) in water, wastewater, soils/sediments, wastes, oils, sludges, and solids. The attached quantitation report lists the target compounds, an example of the retention time order of each target compound, the quantitation and confirmation ions of the target compounds, and internal standard assignments.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria for each target compound are listed in Section 5 of the current revisions of the STL Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 Aqueous samples may be purged at ambient conditions (recommended) or at 40C (optional). Five to twenty-five milliliter aliquots of the sample may be purged. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

2.3 Low-level (nominally <1mg/kg) soil samples are purged at 40C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards are purged under the same conditions.

2.4 High level soils (nominally >1mg/kg) and waste samples are extracted with methanol-1mL of methanol per gram of sample. An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

2.5 This method is based on the guidance in SW-846 Methods 8260B and 5035.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedure that you do not understand or that will put yourself or others in a potentially hazardous situation.

3.2 Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, eye protection, or other equipment should be used. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

3.4 The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed a minimum of every three months and disposed of in accordance with STL-SL SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.

4.2 The volatiles lab must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator blanks should be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in STL-SL SOP AN70: *Segregation of Low and High Concentration Volatile and Semivolatile Samples.*

4.3 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-terachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the lab can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.

4.4 The analysis of highly contaminated samples (>1mg/L or >1mg/kg) can affect succeeding analyses. Carry-over can occur when low concentration samples are analyzed after high concentration samples. Trap replacement and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when carryover is suspected to demonstrate that the system is free from contamination.

4.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If contamination of the fittings is suspected, the fittings may be heated at 105 C for one hour or replaced.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

5.1 Liquid samples are collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are acidified at the time of collection with about 0.10mL of concentrated HCl per 40mL of sample. The acid prevents the biological degradation of the aromatic compounds and prevents the dehydrohalogenation of some of the chlorinated alkanes. The sample must be iced at the time of collection and refrigerated at 4C (less than 6C with no frozen samples) in the lab until analysis.

Check each sample vial at the time of receipt for the presence of "bubbles". If the bubbles are less than 3mm in diameter, the vial is acceptable. If the bubble is greater than 3mm, use another vial. Notify the department supervisor or project manager if there are no acceptable vials for analysis.

A "sacrificial" vial or the vial used for screening analysis is used to check the sample pH. If the sample pH is greater than 2, notify the department supervisor or project manager. If directed by supervisor or project manager, hydrochloric acid may be added through the septum to bring the pH <2. Do not add more than 400uL (0.40mL) of 1:1 HCl to a VOC vial. If pH cannot be adjusted to <=2 without destroying the integrity of the sample, the sample must be analyzed within 7 days of collection.

The holding time for samples preserved with HCl is 14 days for all target compounds. The holding time for un-preserved samples is 7 days.

5.2 Soils: Soils are routinely collected in duplicate in Encore samplers. A "bulk" sample is also routinely collected in a 125-mL jar fitted with a Teflon-lined cap. The bulk sample can be used for the methanol extraction if the concentration of the sample collected in the Encore exceeds the working range of the analytical system.

Soils collected in Encore samplers must be analyzed within 48 hours of collection or must be transferred within 48 hours of collection to sealed vials containing sodium bisulfate solution or methanol. If the sample contains high levels of carbonates, the sample is preserved with water and frozen until the time of analysis. The procedure for preparing soil samples is given in Section 9.2.

The hold time of the preserved sample is 14 days from the date of collection. The hold time for frozen samples is 14 days from the date of collection.

5.3 High level soil and waste samples are collected in glass containers (usually 125-mL clear glass) equipped with Teflon-lined caps. Soil samples may also be submitted as core samples contained in Encore samplers, metal or plastic "tubes", or in 40-mL VOA vials. The samples are iced at the time of collection and stored at 4C (less than 6C with no frozen samples). The holding time for soil and waste samples subjected to methanol extraction is14 days from date of collection; that is, the extraction and analysis must be completed within 14 days of collection.

5.4 TCLP leachate samples are collected with no headspace in Tedlar bags or syringes. The leachate samples are acidified at the time of collection (after the leaching procedure) with about 0.10mL of concentrated HCl per 40mL of sample and stored at 4C (less than 6C with no frozen samples) from the time leaching is completed until the analysis. The acidified leachate sample must be analyzed within 14 days of the leaching procedure. If the sample is not acidified, the leachate must be analyzed within 7 days of the leaching procedure.

NOTE: Samples that are suspected of having very high concentrations of VOC should be segregated from the "routine" samples and stored in a manner that will minimize sample and laboratory contamination. See STL-SL SOP AN70. If possible, keep the field QC in the same storage refrigerator as the samples.

6.0 APPARATUS AND MATERIALS

The apparatus and materials listed in this section may vary from lab to lab. The items listed are to give guidance and to provide a general overview of the equipment employed in this analysis.

6.1 Mass spectrometer: equipped with a capillary direct interface and a split/splitless injector or molecular jet separator

6.2 Gas chromatograph, compatible with the MS and purge and trap systems. If the GC is equipped with an injector that is operated in the split mode, the exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device. The carbon traps should be changed a minimum of every three months.

6.3 Purge and trap device Tekmar 3000 Liquid Concentrator or equivalent

6.4 Supelco Vocarb 3000 trap or equivalent, Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

6.5 Archon soil analyzer for low level soils, compatible with Tekmar purge and trap instruments. The instrument must be capable of automatically adding water and internal standard to the container while maintaining the septum seal, heating the sample to 40C, and spinning the stir bar to mix the sample during the purging step.

6.5 Data System compatible with the analytical system

6.6 Microsyringes: 10ul, 25ul, 50ul, 100ul, 250ul, 500ul, 2.5mL

6.7 Gastight syringe: 5mL, 25mL with luerlock tip

6.8 Volumetric flasks: 1.0mL, 10mL, 100mL

6.9 Recommended Columns

J&W DB-624: 60m x 0.32mm ID, 1.8um film
J&W DB-624: 20m x 0.18mm ID, 1.8um film

7.0 REAGENTS

Reagents must be tracked in accordance with STL-SL SOP AN44: *Reagent Traceability.*

7.1 Reagent water-free of volatile contaminants (obtained by purging with inert gas or carbon filtration)

7.2 Methanol-Burdich and Jackson, Purge and Trap grade

7.3 Sodium bisulfate-reagent grade. This salt is hydroscopic and should be stored in a dessicator.

7.4 Soil preservation solution- Slowly add, while stirring, 200g of sodium bisulfate to a 1.0-L volumetric containing about 700mL of reagent water. After the salt has dissolved, dilute to volume with reagent water, transfer to a storage container, and store the solution in an area free from VOC-especially water-soluble solvents such as acetone. The reagent should be tested prior to use by the analysis of a blank containing 5mL of the solution. The reagent is acceptable if it meets the same criteria as a method blank.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all stock solutions or neat compounds. All preparation steps must be in accordance with STL-SL SOP AN41: *Standard Material Traceability.*

8.1 Preparation of **Stock Standards** from Neat Compounds

The lab should attempt to obtain a certified primary standard or secondary standard before preparing stock standards from neat materials. If primary stock standards must be prepared in-house, the target concentration range is from 2000ug/mL to 10000ug/mL. SL-SOP AN43: *Standard Preparation* gives the general instructions for the preparation of the stock solutions from neat materials.

8.2 Preparation of the **Working Standard** from Stock Standards

The working standard is prepared from the primary stock standards that are either prepared from neat compounds or purchased as certified solutions. The working standard contains one or more of the target compounds at a concentration suitable for preparing the calibration standards, generally 10-200ug/mL. A known volume of the working standard is then added to a known volume of reagent water to make the calibration standard.

The standards and standard concentrations listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document the standard preparation procedures in the standard traceability log.

8.3 Preparation of the **Calibration Standards** from the Working Standards

The calibration standards are the standards that are analyzed on the instrument. The calibration standard is made by adding a known volume of the working standard to a known volume of reagent water. The instrument must be calibrated using a minimum of five calibration standards. The lowest level standard must be at the reporting limit and the rest of the standards will define the working range of the analytical system.

8.3.1 Add 5.0mL of reagent water to a 5mL-glass syringe or 25ml of reagent water to a 25-ml glass syringe.

8.3.2 Add a known volume of the working standard to 5.0mL or 25ml of reagent water.

NOTE: The calibration standards for the low level soils are prepared using the same procedures as for the 5mL water purge except that the standards are purged at 40C. The lab has the option of using blank sand in the calibration standards.

The calibration standards listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document these standard preparation procedures in the standard traceablity log. A 5mL-purge volume may be used for low level (nominal RL of 1ug/L) if the instrument has sufficient sensitivity to detect the targets and the calibration criteria is met.

9.0 SAMPLE PREPARATION

Composite samples can be prepared using the guidance provided in STL-SL-SOP AN70.

9.1 **Aqueous samples** are analyzed directly by purge and trap/GC-MS. No sample preparation is necessary except to homogenize the sample prior to subsampling. The pH of liquid samples is checked and recorded prior to analysis to determine if the sample has been properly preserved.

9.2 Preparation of Soil Samples (5035)

9.2.1 Remove the Encore samples and the bulk sample from the storage area.

9.2.2 Test an aliquot of the bulk sample for the presence of carbonates.

- Transfer 5g of sample from the bulk sample to a 40mL vial..
- Add 5ml of the sodium bisulfate solution and shake the vial .
- If the sample exhibits effervescence, the Encore samples should be preserved as described above using 5mL of volatile-free water in place of the sodium bisulfate solution and placed in a freezer at −10C. The analytical hold time for frozen samples is 14 days from collection.
- If no effervescence is noted, the Encore samples may be preserved with 5mL soil preservation solution.

9.2.3 Add a stir bar to a vial and weigh the vial and record its tare weight(or tare the vial and stir bar weight by pressing the autotare button).

9.2.4 Transfer the sample from the Encore sampler to the tared vial and record the weight of the sample log.

If the sample effervesced during the carbonate test (9.2.2), add 5.0mL of reagent water and freeze at −10C. The hold time is 14 days from collection.

If not, add 5.0mL of the soil preservation solution, seal the vial, and store the sample at 4C until the time of analysis. The preserved sample must be analyzed within 14 days of collection.

NOTE: A preparation blank is prepared when Encore samples are transferred. The preparation blank contains the same reagents as the samples-either 5mL of reagent water or 5mL of soil preservation solution.

9.3 A methanol extraction is prepared when the concentration of the target compounds (by direct purge) exceeds the working range of the calibration curve. The bulk sample, collected in the 125-mL sample container, can be used to prepare the methanol extraction. Carry out the preparation quickly to minimize the loss of volatiles.

-Mix the sample with a stainless steel spatula and transfer 10g (+/- 0.5g) to a glass vial.

-Add 8uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 10g and 100% percent solids, is calculated:

$$Ct(ug/kg,dw) = \frac{0.008mL \otimes 2500ug/mL}{0.010g \otimes solids} = 2000ug/kg,dw$$

-Shake the sample for two minutes. Allow the solvent to separate from the solids portion of the sample and transfer a 1-2mL aliquot of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

-For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The lab control standard is prepared by adding 8uL of the surrogate spiking solution and 8uL of the matrix spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The matrix spikes are prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) and 8uL of the matrix spiking solution (2500ug/mL) to 10-g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for two minutes. Analyze 125uL of the extract or a smaller volume if the VOC concentration is high.

-Add 125uL of the extract (or a smaller volume if the VOC concentration exceeds the linear range of the system with 125uL) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

9.4 Methanol Extraction for Wastes

Carry out the preparation quickly to minimize the loss of volatiles.

9.4.1 Mix the sample with a stainless steel spatula and transfer 1g (+/- 0.2g) to a glass vial.

9.4.2 Add 10uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. If the sample is completely soluble in the methanol, dilute to a final volume of 10mL. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 1.0g , is calculated:

$$Ct(ug/kg) = \frac{0.010mL \otimes 2500ug/mL}{0.0010g \otimes solids} = 25000ug/kg$$

9.4.2 Shake the sample for one minute. Allow the solvent to separate from the solids portion of the sample and transfer 1mL to 2mL of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) to 10mL of purge and trap grade methanol. Assume a sample weight of 1.0g. Analyze 100uL of the extract.

The lab control standard is prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 100uL of the extract.

The matrix spikes are prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 1g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for one minute.

Add 100uL of the extract (or a smaller volume) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

NOTE: Waste samples may require significant dilution prior to analysis.

10.0 PROCEDURE

The following instrument conditions are recommended. The actual conditions may vary due to differences in instrumentation. The lab must document the instrument conditions in the maintenance log, the data system, or on the analysis log.

10.1 Instrument Conditions

10.1.1 GC Conditions
GC conditions may vary according to the environment and condition of each instrument. The lab must document the instrument conditions to assure consistent results and to aid in trouble-shooting the analytical system. Each lab is responsible for assuring that the conditions necessary to achieve adequate separation and sensitivity of the target analytes are maintained.

10.1.1.1 Example GC temperature program

Initial column temperature: 35 C for 3 minutes
Column temperature program 1: 20C per minute
Intermediate column temperature: 70C for 4 minutes
Column temperature program 2: 10C per minute
Final column temperature: 200C for 5.25 minutes

10.1.1.2 Column flow: Approximately 5-10mL/minute helium with a make-up of 20-25mL/minute helium. Total flow into the jet separator should be about 30mL/minute. The vacuum gauge on the jet separator will read about 0.5Torr.

If no jet separator is used and the column is plumbed directly into the source, the column flow should be adjusted to 0.5-1.0ml/min and a split ratio (desorb to column flow) of about 40:1 established. Smaller bore capillary columns (0.18 to 0.32mm) are required if the column is plumbed directly into the source

10.1.1.3 Mass Spectrometer and interface parameters

Jet separator temperature: 240C
Mass spectrometer interface: 240C
Mass spectrometer source temperature: factory set at 300C
range: 35-300amu, with a minimum scan cycle of 1 scan per second

10.1.2 Purge and Trap Conditions
The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

10.1.2.1 "Three ring trap"-charcoal, Tenax, silica gel
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 180C
Bake time: 8 minutes at 225C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.1.2.1 VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 225C
Bake time: 8 minutes at 250C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets-ketones, ethers, bromoform-may be low and the reporting limit may not be achieved on a routine basis.

10.2 BFB Tune Check

10.2.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each 12-hour clock as a check on the "**tune**" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage. If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

10.2.2 Evaluation of the 4-BFB peak.

10.2.2.1 The chromatogram should exhibit acceptable baseline behavior and the 4-BFB peak should be symmetrical. A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

10.2.2.2 The spectrum of the 4-BFB must meet the criteria listed in the attached SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 5 scans from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.

10.2.2.3 The following records must be kept for each 4-BFB analysis that meets the criteria:
- the date, time, and data file of the analysis
- a spectrum of the scan or averaged scans
- a tabulation of the ion abundances of the scan

10.2.2.4 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , or other anomalies.

10.2.2.5 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

10.3 Initial Calibration

After the 4-BFB criteria has been met, the initial calibration standards are analyzed. Prepare the initial calibration standards according to the example recipes in the SOP appendices or lab-specific recipe. The lab must document the "recipe" used to prepare the calibration standards. The lowest level calibration standard must be at or below the routine RL and the other calibration standards will define the working range of the system.

10.3.1 Remove the plunger from the syringe and fill the barrel to overflowing with reagent water (syringe valve in the "red" position).

10.3.2 Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL or 25mL)

10.3.3 Briefly remove the syringe valve and inject the standards and internal standards into the syringe.

NOTE: Use the internal standard (IST) mix when preparing the calibration standards for analysis. The surrogates are already included in the standard mixes.

10.3.4 Load the standard(s) onto the purge and trap device and begin the analysis. All pertinent information concerning the standards must be recorded on the analysis log. The standards must be clearly identified and traceable to the preparation steps.

NOTE: The standards for low-level soil samples are prepared in the same manner as the 5mL standards. The standards for the low-level soils are purged at 40C. The lab has the option of using blank sand or soil in the calibration standards and the blank in the low level soil analysis.

10.3.5 After the acquisition has taken place, evaluate the calibration standards to ensure that each target compound, surrogate, and internal standard has been correctly identified. The analyst must be careful to complete this step before proceeding.

10.3.6 After each target compound, surrogate, and internal standard has been correctly identified, the relative response factor for each target compound and surrogate is calculated using the data system or using a PC spreadsheet as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where
Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (see the attached quantitation report for a list of the compounds that are associated with the various internal standards)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug on-column)
Cis = concentration or mass on-column of the internal standard (ug/L or ug/kg OR ng or ug on-column)

The average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$RRFavg = \frac{RRF1 + RRF2 + ... + RRFn}{n}$$

where n = number of calibration levels

Calculate the standard deviation (SD) for the target compounds and surrogates at all calibration levels:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RFi - RFavg)^2}{n-1}}$$

where
Rfi = response factor of a target compound in the individual calibration level
Rfavg = average response factor
n= number of calibration levels

10.3.7 Calculate the relative standard deviation (% RSD) of the calibration levels for each target:

$$\% RSD = \frac{standard\ deviation}{RRFavg} \otimes 100$$

10.3.8 The results of the initial calibration are evaluated against the Calibration Check Compound (CCC) criteria and the System Performance Check Compound (SPCC) criteria, which are listed below. The CCC and SPCC criteria must be met before samples can be analyzed.

Calibration Check Compounds – CCC Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

10.3.9 After the initial calibration criteria (CCC and SPCC) have been met, each target is evaluated for linearity.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug)
Cis = concentration of the internal standard (ug/L or ug/kg OR ng or ug)

If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.. Regression curves may be forced through zero but it is recommended that the curve be evaluated without forcing through zero first and then with the curve forced through the origin. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3.10 After the initial calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP or other specified QAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compounds must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of samples can take place.

If the method blank repeatedly fails to meet the criteria, contact the immediate supervisor to determine the cause of the problem and to determine a course of action. This action may include re-cleaning the sparging tubes (with soap, hot water, and methanol), purging the effected autosampler ports with heated methanol, flushing the purge and trap ALS concentrator with methanol, replacing the trap, changing the transfer line, and changing the column. A method blank is then analyzed after taking the corrective action to demonstrate that the contamination has been eliminated. Once the system is determined to be free from contamination, sample analysis may begin. Method blanks may be required after the analysis of samples that contain very high levels of VOC.

10.4 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of 50ng of 4-BFB. This criteria must be met before the analysis of the calibration check standards can take place.

10.4.1 After the tune criteria has been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a nominal concentration of 50ug/L-kg for 5ml/5g samples and 10ug/L for 25mL with ketones and poor purgeables at higher concentrations. The CCC and SPCC criteria (Section 10.3.8) must be met before the analysis of the method blank and samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration
Cccv = measured concentration using the selected quantitation method

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If this criteria is not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.4.2 The calibration standard (CCV) must also be evaluated for internal standard retention time and response.

If the retention time of any internal standard changes by more than 30 seconds from the retention times of the internal standards in the initial calibration, the analytical system must be inspected for problems and corrective action instituted.

If the extracted ion current profile (EICP) area for any of the internal standards changes by more than a factor of two (-50% to +100%) from the last calibration check standard, the analytical system must be inspected for problems and corrective action instituted. If the CCV is the first one after the initial calibration, compare the ISTD response to the corresponding level in the ICAL.

10.4.3 After the continuing calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compound must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of client samples can take place.

10.5 Aqueous Sample Analysis-5.0mL to 25mL

The analyst must use the same volume as was used for the calibration standards-if a 5mL sample is used, it must be quanted off of the 5mL calibration curve; if a 25ml sample is used, it must be quanted off of the 25mL calibration curve. Samples are analyzed only after the tune criteria, the calibration (initial or continuing) criteria has been met, and the method blank criteria has been met. See the SOP Summary for the analytical sequence.

10.5.1 Remove the samples to be analyzed from the refrigerator and allow the samples to come to ambient temperature.

10.5.2 Put on a pair of gloves before transferring the sample from the vial to the syringe. The sample is most likely preserved with acid or may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. ***Gloves must be worn when handling samples.***

10.5.3 Mix the contents of the vial by inverting the vial several times. Check to see if there are air bubbles present in the sample. If air bubbles are present, use another vial if available. Make a note on the analysis log if the sample used contained bubbles and notify the supervisor and/or the project manager.

10.5.5 Remove the plunger from the glass syringe. Attach a syringe valve to the syringe Luer-tip to prevent sample from spilling out of the syringe when sample is added.

10.5.5 Open the vial of the well-mixed sample and gently pour the sample into the syringe barrel. The sample should fill the barrel of the syringe and overflow to allow trapped air bubbles to escape.

10.5.6 Replace the plunger into the syringe barrel. Try not to let air bubbles get into the barrel. If air bubbles are present, turn the syringe up, open the syringe valve , and expel the air while adjusting the volume to 5.0mL or 25mL. If no air bubbles were trapped, adjust the syringe to volume.

NOTE: For TCLP leachate samples, use 1.25mL of sample (1:4 dilution).

10.5.7 Open the syringe valve and inject the internal standard/surrogate (ISSU) mix into the sample.

10.5.8 Transfer the sample from the syringe to the purge and trap device. Record all of the sample identification information on the analysis log. Check the pH of the sample with pH paper and record the pH on the instrument log or other appropriate log.

10.5.9 Analyze the samples using the purge and trap and GC/MS conditions used for the initial and continuing calibration standards.

10.5.10 Determine the concentration of the samples and QC items. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

A dilution is made when a volume of the sample is mixed with the reagent water to a final volume of 5.0mL or 25ml,depending on which curve is being used. The dilution factor is calculated by dividing the volume of sample into the volume used for the calibration curve.

$$DF = \frac{final\ volume\ of\ dilution(mL)}{volume\ of\ sample\ used(mL)}$$

For example, if 1.0mL of sample is diluted to final volume of 5.0mL, the dilution factor is 5. (5.0/1.0 = 5). If 1.0mL of sample is diluted to a final volume of 25mL, the dilution factor is 25 (25/1=25).

The following table gives some dilution factors:

Volume of Sample (mL)	Volume of Reagent Water (mL)	Final Volume (mL)	Dilution factor
5.0	0	5.0	1
2.5	2.5	5.0	2
1.0	4.0	5.0	5
0.5	4.5	5.0	10
0.10	4.9	5.0	50
25.0	0	25.0	1
5.0	20.0	25.0	5
2.5	22.5	25.0	10
1.0	24.0	25.0	25
0.50	24.5	25.0	50
0.10	24.9	25.0	250

NOTE: The same volume of internal standard/surrogate mix (ISSU) is added to the dilution as was added to the undiluted sample.

10.6 Low Level Soil Samples by Heated Purge and Trap (Method 5035)

The soil analytical system is calibrated using the same concentrations as the 5mL purge. The tune, initial and continuing calibration criteria, and the method blank criteria must be met before samples are analyzed. Standards and QC items must be analyzed under the same heated purge and trap conditions.

Remove the samples to be analyzed (Section 9.2) from the refrigerator or freezer and allow the sample to come to ambient temperature. Inspect the vial for cracks or obvious breaches in the septum. Load the samples on to the soil-purging unit and analyze according to the sequence described in Appendix B.

Liquid field QC for soils (trip blank, field blank, etc.) should be analyzed with the associated soil samples, using the same preparation and analytical procedures, including the heated purge. Report the results for liquid trip blanks as ug/L.

10.7 Analysis of Methanol Extracts of Soils and Wastes

The methanol extraction is used when the concentration of one or more target compounds exceeds the linear range of the low-level purge technique (>1000ug/kg). or if the concentration of VOC in the soil or waste samples is high. Samples are analyzed only after the 4-BFB criteria, the calibration criteria (initial and continuing), and the method blank criteria has been met. Medium level soil extracts are quanted using the ambient purge calibration curve. Sample preparation steps are included in Section 9.

10.7.1 Remove the plunger from the 5.0-mL syringe and fill the barrel to overflowing with reagent water(syringe valve in the "red" position). Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL)

10.7.2 Briefly remove the syringe valve and inject the sample extract and 5uL of the internal standard (IST) solution into the syringe. Use 125ul of the extract for soils and 100uL of the extract for wastes. Smaller aliquots are used if the concentration of target analytes exceed the working range of the system.

NOTE: Use the internal standard (IST) mix when preparing the medium level samples. Recall that the surrogates have already been added to the sample during the methanol extraction step (Section 9).

10.7.3 Load the sample on to the purge and trap device and begin the analysis. All pertinent information concerning the samples must be recorded on the analysis log. The samples must be clearly identified and traceable to the extraction log. These conditions must be the same as was used for the initial and continuing calibration standards-ambient purge for aqueous samples.

10.7.4 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, a smaller aliquot of the methanol extract is reanalyzed to bring the highest target within the upper half of the calibration curve. Follow the guidelines in Section 10.4.10 for reporting dilutions.

NOTE: It is possible to dilute the surrogates in the sample extract below the linear range of the calibration curve. The minimum extract aliquot that can be used to provide a quantifiable result for the surrogates and matrix spikes is 0.0025mL (2.5uL).

SOIL: 10g to 10mL MeOH	WASTES: 1g to 10mL MeOH	Surrogates- Theoretical ng on-column
125uL(0.125mL)	100uL (0.100mL)	250
62.5uL(0.0625mL)	50uL(0.050mL)	125
25uL(0.025mL)	25uL(0.020mL)	50
12.5uL(0.0125mL)	10ul(0.010mL)	25
2.5uL(0.0025mL)	2.0uL(0.0020mL)	5.0-quantiation limit
<2.5uL(0.025mL)	<2.0uL(0.0020mL)	<5.0ng- below the quantitation limit-diluted out

NOTE: Some instrument quantitation limits may be higher than the limit listed in the table. The volume of extract should be adjusted accordingly.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis of Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1 Two criteria must be met in order to identify a target compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{\textit{retention time of the target compound}}{\textit{retention time of the associated internal standard}}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. These ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification. Tentative identifications of non-targets will be made only by analysts having completed the training specified in the training schedule.

11.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.2.2 The relative intensities of the major ions should agree within +/-30%.

11.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult other analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculation is

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes DF$$

where
Cis = concentration of the internal standard, ug/L
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
DF = dilution factor

Soils by Heated P/T

$$TIC\,(ug/kg, dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{5.0g}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
W = weight of sample analyzed, g
solids = decimal equivalent of percent solids

Soils by Methanol Extraction

$$TIC\,(ug/kg, dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{Vcal}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = decimal equivalent of the percent solids(percent solids/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

where
Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

11.3 Calculations for Samples-Internal Standard Technique

Aqueous Samples- relative response factor :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes DF$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
DF = dilution factor

Aqueous Samples: regression curve

$$concentration(ug/L) = concentration(curve) \otimes DF$$

where
DF = dilution factor

The reporting limit (RL) is calculated:

$$RL(ug/L) = RLqap \otimes DF$$

where
DF = dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

Soils by Heated P/T- relative response factor :

$$concentration(ug/kg, dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{5.0g}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/kg)
RRFavg = average response factor of the compound being measured
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

Soils by Heated P/T: regression curve

$$conc(ug/kg, dw) = Ccurve(ug/kg) \otimes \frac{5.0g}{(W)(solids)}$$

where
Ccurve = concentration from curve(ug/kg)
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The reporting limit (RL) is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

Methanol Extraction Soils and Wastes- relative response factor

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{Vcal}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = (percent solids)/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

Methanol Extraction of Soils and Solids- regression curve:

$$conc(ug,kg,dw) = Ccurve(ug/L) \otimes \frac{Vcal}{(W)(solids)}$$

where
Vcal = volume that calibration curve is based on (0.005L or 0.025L)
W = weight of sample added to the reagent water (g)-defined above

The reporting limit (RL)is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the reagent water (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in Section 3.1.3 of STL-SL SOP AN02: *Analytical Batching*. Note that the method blank for liquid samples and low-level soils is clock-specific and that the method blank for medium level soil samples is extraction batch-specific.

STL-SLSOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. This criteria is summarized in the attached 8260 SOP Summary.

STL-SL SOP AN02 also contains the calculations for accuracy and precision and the calculations for the theoretical concentrations of surrogates, lab spikes, and matrix spikes.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration is the preparation and analysis of spiked reagent water. Section 8.3 of EPA Method 8260A gives the general procedure for the performance of the IDOC and Table 6 of EPA Method 8260A gives the acceptance criteria for the accuracy and precision.

12.3 Method Detection Limit

The method detection limit is determined in accordance with STL-SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. Section 10 of the STL-SL QAPs provide guidance on preventive maintenance.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time. See instrument manufacturers' manuals for guidance on locating and repairing instrument problems.

15.0 REFERENCES

1. *Savannah Laboratories' Comprehensive Quality Assurance Plan* and *Savannah Laboratories' Corporate Quality Assurance Plan*, current revisions.

2. Method s 5035, 8000B, and 8260B. *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846.including Update III* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Appendix A

VOLATILES BY GC/MS WORKING STANDARDS -EXAMPLE

These standards can be used to prepare the working standards for EPA Method 8260 to report the TCL (target compound list) compounds and the extended list of target compounds generally associated with EPA 8260. The standards are prepared in purge and trap grade methanol and are stored at 4C with minimum headspace.

Working Standard 1 (TCL WS-1)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #2	2000	12.5	25
VOA Cal #3	2000	12.5	25
VOA Cal #4	2000	12.5	25
1,2,-DCB	5000	5.0	25
1,3-DCB	5000	5.0	25
1,4-DCB	5000	5.0	25
2-CEVE	1000	125	125

Working Standard 2 (TCL WS-2)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #1	5000	25	125
8260 Surrogates	2500	10	25

Working Standard for GASES (TCL GASES)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
502.2 Cal 1	2000	12.5	25

Appendix A

Working Standard 3 (8260 WS-3)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
8260 Custom Mix #1	200	125	25
8260 Custom Mix #2	200	125	25
1,1,2,2-Tetrachloroethane	2000	12.5	25

Appendix A

Internal Standard (8260 ISTD)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50

Internal Standard/Surrogate (8260 ISSU)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50
8260 Surrogate	2500	20	50

Tune Evaluation Standard (4-BFB)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
4-BFB	5000	10	50

Matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
Matrix Spiking Solution	2500	20	50

TCLP matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
TCLP Spiking Solution	2000	16	125

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS - EXAMPLES

The following calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	TCL-1 *	TCL-2 *	TCL-3 *	TCL-4 *	TCL-5 *	TCL-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	8260-1 *	8260-2 *	8260-3 *	8260-4 *	8260-5 *	8260-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

CONCENTRATIONS OF THE CALIBRATION STANDARDS-5.0mL OR 5.0g

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
TCL-1,8260-1	5ug/l-kg	25ug/l-kg
TCL-2,8260-2	10ug/l-kg	50ug/l-kg
TCL-3,8260-3	25ug/l-kg	125ug/l-kg
TCL-4,8260-4	50ug/l-kg	250ug/l-kg
TCL-5,8260-5	100ug/l-kg	500ug/l-kg
TCL-6,8260-6	200ug/l-kg	1000ug/l-kg

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS-25mL Purge Volume-EXAMPLES

These calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The standards are based on a volume of 25mL to achieve lower quantitation limits for the target compounds. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	25TCL -1*	25TCL -2*	25TCL-3*	25TCL -4*	25TCL-5*	25TCL-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	258260-1*	258260 -2*	258260-3*	258260-4*	258260-5*	258260-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

CONCENTRATIONS OF THE CALIBRATION STANDARDS

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
25TCL-1,25-8260-1	1.0ug/l	5.0ug/l
25TCL-2,25-8260-2	2.0ug/l	10ug/l
25TCL-3,25-8260-3	5.0ug/l	25ug/l
25TCL-4,25-8260-4	10ug/l	50ug/l
25TCL-5,25-8260-5	20ug/l	100ug/l
25TCL-6,25-8260-6	40ug/l	200ug/l

Appendix B
8260 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Container	Hold Time
Aqueous	None; 4C	40mL no headspace	7 days
	HCl pH<2; 4C	40mL-no headspace	14 days
Soil/solid(low level)	Iced at collection; 5mL sodium bisulfate added upon arrival in lab; store at 4C	5-g Encore Sampler	14 days
Soil/solid(low level) -high carbonates	Iced at collection; 5mL water added upon arrival in lab; store at −10C	5-g Encore Sampler	14 days
Soil/solid(high level)	None; 4C	Glass 125mL	14 days
TCLP	HCl pH<2; 4C	Tedlar bag or syringe	14 days

*storage temperature is 4C with a control criteria of less than 6C with no frozen samples

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
4-BFB 50ng on column Clock starts at injection	4-BFB 50ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification (50ug/L or 50ug/kg)) RL Standard-low point on cal curve (if necessary)
Method blank	Method blank
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

See SL SOP AN02, Section 3.1.3, for the batch/clock options for LCS and MS/MSD.

Recommended Internal Standards:
1,2-dichloroethane-d4; 1,4-difluorobenzene; chlorobenzene-d5; 1,4-dichlorobenzene-d4

Surrogates/System Monitoring Compounds:
dibromofluoromethane; toluene-d8; 4-bromofluorobenzene

LCS/MS: CQAP Subset:
1,1-dichloroethene; benzene; trichloroethene; toluene; chlorobenzene

Appendix B
8260 SOP SUMMARY

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)	
m/e	**Abundance Criteria**
50	8.0-40.0% of mass 95
75	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	50-120%% of mass 95
175	4.0-9.0% of mass 174
176	93.0-101.0% of mass 174
177	5.0-9.0% of mass 176

(1) *8260 criteria taken from CLP OLMO4.0 (January 1998)

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	**Continuing Calibration**
Less than or equal to 30% RSD	Less than or equal to 20% difference or drift from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

See Sections 10.3 and 10.4 for ICAL and CCV linearity checks and criteria.

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
MS Tune Check - 50ng 4-BFB	Before initial and continuing calibration standards - every 12 hours	Mass abundances within method acceptance criteria	-Evaluate chromatogram and spectrum - Reanalyze - Retune MS and reanalyze - Remake standard and reanalyze - Perform instrument maintenance and reanalyze
Initial Calibration – minimum five point curve with lowest point at or below the Reporting Limit (RL)	Initially; after major instrument maintenance; whenever continuing calibration check fails. Prior to analysis of method blank and samples	Method criteria for CCC/SPCC (see -Calibration Acceptance Criteria – Table presented earlier in this document)	- Evaluate chromatograms, spectra, and integrations - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument maintenance and recalibrate
Continuing Calibration check - midpoint standard	Every 12 hours before analysis of method blank and samples	Method criteria for CCC/SPCC (see Calibration Acceptance Criteria - Table presented earlier in this document)	- Evaluate chromatogram, spectra, integrations - Reanalyze standard - Remake and reanalyze standard - Recalibrate - Perform instrument maintenance and recalibrate
Method Blank	Every 12 hours (per clock) before sample analyses	All reported targets <RL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

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Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Lab Control Sample (LCS) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Matrix Spike/Matrix Spike Duplicate (MS/MSD) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogates	All samples, blanks, LCS, MS	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Internal Standard Area	Evaluate all standards and samples	-Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence -Retention time of internal standard must be +/-30 seconds from internal standard in initial calibration -Areas in samples should be evaluated for gross error . Consult supervisor.	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Reporting Limit Standard -1x to 2x the RL	(Optional) Daily. Required for Florida DEP	Detected with reasonable response	-Evaluate chromatogram, spectra, and integrations -Reanalyze -Remake standard and reanalyze -Retune and recalibrate -Perform instrument maintenance and recalibrate
Initial Demonstration of Capability	Per analyst	Method criteria	-Reanalyze targets that do not meet criteria
Method Detection Limit (MDL)	See STL-SL SOP CA90	See STL-SL SOP CA90	-Reanalyze and re-evaluate

Appendix C
EXAMPLE QUANTITATION REPORT

-quantitation ions
-internal standard and target compound association

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m
Quant Method : ISTD Target Version : 3.50
Last Update : 12-Aug-1999 11:49 Number of Cpnds : 106
Data Type : MS DATA

Global Integrator : HP RTE

Chromat Events	Values
Initial:Thresh Units	0.000000
Initial:Area Thresh	200.000000
Initial:Max Peaks	100.000000
Initial:Bunching	2.000000
Initial:Smoothing	1.000000
Initial:Start Thresh	0.057000
Initial:Stop Thresh	0.142000
Initial:Baseline Reset	5.000000
Initial:Set Valley	100.000000

Compound	RT	RT Window	RF	Mass
1 DICHLORODIFLUOROMETHANE	2.291	1.842-2.740	6.813e-01	85.00
	2.291	1.842-2.740		87.00
	2.291	1.842-2.740		101.00
2 CHLOROMETHANE	2.589	2.140-3.038	1.054e+00	50.00
	2.595	2.146-3.044		52.00
3 VINYL CHLORIDE	2.729	2.280-3.178	1.429e+00	62.00
	2.723	2.274-3.172		64.00
4 BROMOMETHANE	3.173	2.724-3.622	3.097e+00	94.00
	3.173	2.724-3.622		96.00
	3.167	2.718-3.616		79.00
5 CHLOROETHANE	3.319	2.870-3.768	7.702e-01	64.00
	3.319	2.870-3.768		66.00
6 TRICHLOROFLUOROMETHANE	3.672	3.223-4.121	1.210e+00	101.00
	3.672	3.223-4.121		103.00
	3.672	3.223-4.121		105.00
7 TRICHLOROTRIFLUOROETHANE(1	3.672	3.223-4.121	1.173e+00	101.00
	3.672	3.223-4.121		151.00
	3.672	3.223-4.121		103.00
8 ACROLEIN	3.988	3.434-4.542		56.00
	3.988	3.434-4.542		55.00
9 ACETONITRILE	4.070	3.516-4.624		41.00
	4.070	3.516-4.624		40.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

Compound	RT	RT Window	RF	Mass
10 1 1-DICHLOROETHENE	4.317	3.868-4.765	1.053e+00	96.00
	4.317	3.868-4.765		61.00
	4.323	3.874-4.772		98.00
11 ACETONE	4.359	3.910-4.808	1.171e-01	58.00
	4.359	3.910-4.808		43.00
12 DIMETHYL SULFIDE	4.557	4.003-5.111		47.00
	4.557	4.003-5.111		62.00
	4.557	4.003-5.111		45.00
13 CARBON DISULFIDE	4.609	4.160-5.057	4.138e+00	76.00
	4.609	4.160-5.057		78.00
14 IODOMETHANE	4.666	4.112-5.220		141.90
	4.666	4.112-5.220		126.90
15 METHYL MERCAPTAN	4.827	4.273-5.381		47.00
	4.827	4.273-5.381		48.00
	4.827	4.273-5.381		45.00
16 ACRYLONITRILE	4.876	4.322-5.430		53.00
	4.876	4.322-5.430		52.00
	4.876	4.322-5.430		51.00
17 METHYLENE CHLORIDE	4.895	4.446-5.343	1.289e+00	84.00
	4.888	4.440-5.337		49.00
	4.888	4.440-5.337		86.00
18 3-CHLORO-1-PROPENE	4.946	4.392-5.500		41.00
	4.946	4.392-5.500		76.00
19 METHYL T-BUTYL ETHER	5.223	4.774-5.672	2.979e-03	73.00
	5.223	4.774-5.672		57.00
20 trans-1 2-DICHLOROETHENE	5.235	4.786-5.684	1.213e+00	96.00
	5.241	4.792-5.690		61.00
	5.235	4.786-5.684		98.00
21 1 1-DICHLOROETHANE	5.734	5.285-6.183	1.785e+00	63.00
	5.734	5.285-6.183		65.00
	5.752	5.303-6.201		83.00
22 VINYL ACETATE	5.771	5.322-6.219	1.308e+00	43.00
	5.771	5.322-6.219		86.00
23 2-BUTANONE	6.446	5.997-6.895	4.896e-01	43.00
	6.446	5.997-6.895		72.00
24 cis-1 2-DICHLOROETHENE	6.458	6.009-6.907	1.297e+00	96.00
	6.458	6.009-6.907		61.00
	6.446	5.997-6.895		98.00
25 2 2-DICHLOROPROPANE	6.458	6.009-6.907	7.527e-01	77.00
	6.458	6.009-6.907		41.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
	26 BROMOCHLOROMETHANE	6.750	6.301-7.199	5.114e-01	127.90
		6.750	6.301-7.199		49.00
		6.750	6.301-7.199		129.90
	27 CHLOROFORM	6.835	6.386-7.284	1.687e+00	83.00
		6.835	6.386-7.284		85.00
		6.841	6.392-7.290		47.00
	28 PROPIONITRILE	6.885	6.331-7.439		54.00
		6.885	6.331-7.439		55.00
$	29 DIBROMFLUOROMETHANE	7.048	6.599-7.497	8.084e-01	112.90
		7.030	6.581-7.479		81.00
		7.048	6.599-7.497		110.90
	30 1 1 1-TRICHLOROETHANE	7.103	6.620-7.586	3.268e-01	97.00
		7.103	6.620-7.586		99.00
		7.109	6.626-7.592		61.00
	31 1 1-DICHLOROPROPENE	7.316	6.833-7.799	3.671e-01	75.00
		7.316	6.833-7.799		110.00
		7.316	6.833-7.799		77.00
	32 HEXANE	7.325	6.771-7.879		57.00
		7.325	6.771-7.879		41.00
		7.325	6.771-7.879		43.00
	33 CARBON TETRACHLORIDE	7.328	6.845-7.811	2.509e-01	116.90
		7.328	6.845-7.811		118.90
		7.328	6.845-7.811		120.90
	34 TETRAHYDROFURAN	7.410	6.856-7.964		42.00
		7.410	6.856-7.964		71.00
		7.410	6.856-7.964		72.00
*	35 1,2-DICHLOROETHANE-d4	7.480	7.031-7.929		65.00
		7.480	7.031-7.929		67.00
		7.492	7.043-7.941		102.00
	36 CHLOROPRENE	7.492	6.938-8.046		53.00
		7.492	6.938-8.046		88.00
	37 1 2-DICHLOROETHANE	7.590	7.106-8.073	2.546e-01	62.00
		7.577	7.094-8.060		49.00
		7.590	7.106-8.073		64.00
	38 BENZENE	7.590	7.106-8.073	1.335e+00	78.00
		7.583	7.100-8.067		50.00
		7.583	7.100-8.067		51.00
	39 METHACRYLONITRILE	7.959	7.405-8.513		67.00
		7.959	7.405-8.513		52.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
*	40 1,4-DIFLUOROBENZENE	8.052	7.569-8.535		114.00
		8.052	7.569-8.535		63.00
		8.058	7.575-8.541		88.00
	41 TRICHLOROETHENE	8.435	7.952-8.918	2.915e-01	129.90
		8.435	7.952-8.918		95.00
		8.429	7.946-8.912		131.90
	42 1 2-DICHLOROPROPANE	8.733	8.250-9.216	3.098e-01	63.00
		8.727	8.244-9.210		76.00
		8.733	8.250-9.216		65.00
	43 ISOBUTANOL	8.883	8.329-9.437		43.00
		8.883	8.329-9.437		41.00
	44 DIBROMOMETHANE	8.904	8.420-9.387	1.437e-01	93.00
		8.904	8.420-9.387		173.80
		8.904	8.420-9.387		95.00
	45 BROMODICHLOROMETHANE	9.104	8.621-9.587	3.080e-01	83.00
		9.110	8.627-9.593		85.00
		9.098	8.615-9.581		129.00
	46 ETHYL ACETATE	9.303	8.749-9.857		43.00
		9.303	8.749-9.857		45.00
		9.303	8.749-9.857		88.00
	47 2-CHLOROETHYL VINYL ETHER	9.506	9.023-9.989	1.457e-01	63.00
		9.512	9.029-9.995		65.00
		9.500	9.017-9.983		106.00
	48 CYCLOHEXANE	9.592	8.945-10.239		56.00
		9.592	8.945-10.239		84.00
	49 cis-1,3-DICHLOROPROPENE	9.749	9.266-10.232	4.493e-01	75.00
		9.749	9.266-10.232		77.00
		9.743	9.260-10.226		110.00
	50 2-HEXANONE	9.950	9.205-10.695	5.456e-01	43.00
		9.950	9.205-10.695		58.00
$	51 TOLUENE-d8	10.163	9.680-10.646	1.031e+00	98.00
		10.163	9.680-10.646		100.00
		10.151	9.668-10.634		70.00
	52 TOLUENE	10.260	9.777-10.743	7.674e-01	92.00
		10.260	9.777-10.743		91.00
		10.254	9.771-10.737		65.00
	53 HEPTANE	10.487	9.840-11.134		43.00
		10.487	9.840-11.134		57.00
		10.487	9.840-11.134		71.00

Savannah Laboratories

COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
	54 trans-1,3-DICHLOROPROPENE	10.558	10.075-11.041	3.927e-01	75.00
		10.564	10.081-11.047		77.00
		10.558	10.075-11.041		110.00
	55 1 1 2-TRICHLOROETHANE	10.844	10.361-11.327	2.219e-01	83.00
		10.850	10.367-11.333		97.00
		10.844	10.361-11.327		99.00
	56 TETRACHLOROETHENE	11.106	10.361-11.851	5.192e-01	163.90
		11.118	10.373-11.863		165.90
		11.112	10.367-11.857		167.90
	57 1 3-DICHLOROPROPANE	11.112	10.629-11.595	4.788e-01	76.00
		11.112	10.629-11.595		78.00
		11.112	10.629-11.595		41.00
	58 4-METHYL-2-PENTANONE (MIBK	11.215	10.732-11.698	1.708e-01	43.00
		11.215	10.732-11.698		57.00
		11.215	10.732-11.698		58.00
	59 DIBROMOCHLOROMETHANE	11.471	10.726-12.216	5.124e-01	128.90
		11.471	10.726-12.216		126.90
		11.477	10.732-12.222		130.90
M	60 1,2-DICHLOROETHENE (total)	11.485	7.016-8.164	1.255e+00	96.00
	61 1 2-DIBROMOETHANE	11.672	11.188-12.155	2.421e-01	107.00
		11.672	11.188-12.155		109.00
	62 METHYL METHACRYLATE	12.235	11.588-12.882		69.00
		12.235	11.588-12.882		41.00
	63 1-CHLOROHEXANE	12.409	11.664-13.154	1.251e+01	41.00
		12.409	11.664-13.154		43.00
		12.409	11.664-13.154		55.00
*	64 CHLOROBENZENE-d5	12.420	11.675-13.165		82.00
		12.420	11.675-13.165		117.00
		12.420	11.675-13.165		119.00
	65 CHLOROBENZENE	12.462	11.717-13.207	1.691e+00	112.00
		12.462	11.717-13.207		77.00
		12.456	11.711-13.201		51.00
	66 1 1 1 2-TETRACHLOROETHANE	12.584	11.839-13.329	5.385e-01	130.90
		12.584	11.839-13.329		132.90
		12.584	11.839-13.329		119.00
	67 ETHYL BENZENE	12.633	11.887-13.378	3.045e+00	91.00
		12.633	11.887-13.378		106.00
		12.627	11.881-13.372		51.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

Compound	RT	RT Window	RF	Mass
68 m,p-XYLENE	12.821	12.076-13.566	1.187e+00	106.00
	12.827	12.082-13.573		91.00
	12.827	12.082-13.573		77.00
69 DIMETHYL DISULFIDE	13.378	12.731-14.025		94.00
	13.378	12.731-14.025		45.00
	13.378	12.731-14.025		79.00
70 o-XYLENE	13.497	12.751-14.242	1.144e+00	106.00
	13.497	12.751-14.242		91.00
	13.503	12.757-14.248		77.00
71 STYRENE	13.515	12.770-14.260	1.885e+00	104.00
	13.515	12.770-14.260		78.00
	13.515	12.770-14.260		103.00
72 BROMOFORM	13.831	13.086-14.576	3.683e-01	172.80
	13.825	13.080-14.570		170.80
	13.831	13.086-14.576		174.80
73 ISOPROPYLBENZENE	14.123	13.137-15.109	3.229e+00	105.00
	14.123	13.137-15.109		120.00
	14.123	13.137-15.109		77.00
$ 74 p-BROMOFLUOROBENZENE	14.391	13.646-15.136	7.764e-01	95.00
	14.385	13.639-15.130		174.00
	14.385	13.639-15.130		176.00
75 1 1 2 2-TETRACHLOROETHANE	14.622	13.877-15.367	7.407e-01	83.00
	14.622	13.877-15.367		85.00
	14.616	13.871-15.361		168.00
76 BROMOBENZENE	14.659	13.673-15.644	7.575e-01	156.00
	14.659	13.673-15.644		77.00
	14.659	13.673-15.644		158.00
77 1 2 3-TRICHLOROPROPANE	14.701	13.715-15.687	2.250e-01	110.00
	14.701	13.715-15.687		112.00
78 n-PROPYLBENZENE	14.841	13.855-15.827	9.029e-01	120.00
	14.841	13.855-15.827		91.00
	14.841	13.855-15.827		65.00
79 2-CHLOROTOLUENE	14.999	14.013-15.985	7.182e-01	126.00
	14.999	14.013-15.985		91.00
	14.999	14.013-15.985		63.00
80 n-BUTYL ACETATE	15.096	13.997-16.195		56.00
	15.096	13.997-16.195		43.00
81 1 3 5-TRIMETHYLBENZENE	15.145	14.159-16.131	2.424e+00	105.00
	15.145	14.159-16.131		120.00
	15.145	14.159-16.131		77.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

	Compound	RT	RT Window	RF	Mass
	82 4-CHLOROTOLUENE	15.182	14.196-16.167	7.548e-01	126.00
		15.182	14.196-16.167		91.00
		15.182	14.196-16.167		63.00
M	83 XYLENE (total)	15.547	15.083-16.231	1.197e+00	106.00
	84 tert-BUTYLBENZENE	15.723	14.737-16.709	2.362e+00	119.00
		15.723	14.737-16.709		91.00
		15.723	14.737-16.709		134.00
	85 1 2 4-TRIMETHYLBENZENE	15.814	14.829-16.800	2.418e+00	105.00
		15.814	14.829-16.800		120.00
		15.814	14.829-16.800		77.00
	86 ETHYL METHACRYLATE	15.843	14.744-16.942		69.00
		15.843	14.744-16.942		41.00
		15.843	14.744-16.942		39.00
	87 sec-BUTYLBENZENE	16.119	15.133-17.104	3.458e+00	105.00
		16.119	15.133-17.104		134.00
		16.119	15.133-17.104		91.00
	88 1 3-DICHLOROBENZENE	16.325	15.340-17.311	1.404e+00	146.00
		16.319	15.334-17.305		148.00
		16.313	15.327-17.299		111.00
	89 p-ISOPROPYLTOLUENE	16.374	15.388-17.360	2.676e+00	119.00
		16.374	15.388-17.360		134.00
		16.374	15.388-17.360		91.00
*	90 1,4-DICHLOROBENZENE d4	16.429	15.443-17.415		152.00
		16.429	15.443-17.415		150.00
		16.423	15.437-17.408		115.00
	91 1 4-DICHLOROBENZENE	16.471	15.486-17.457	1.431e+00	146.00
		16.471	15.486-17.457		148.00
		16.478	15.492-17.463		111.00
$	92 1,2-DICHLOROBENZENE D4	17.104	16.118-18.090	1.403e+00	150.00
		17.104	16.118-18.090		152.00
		17.104	16.118-18.090		115.00
	93 n-BUTYLBENZENE	17.122	16.136-18.108	2.737e+00	91.00
		17.122	16.136-18.108		92.00
		17.122	16.136-18.108		134.00
	94 1 2-DICHLOROBENZENE	17.141	16.155-18.126	1.237e+00	146.00
		17.141	16.155-18.126		148.00
		17.135	16.149-18.120		111.00
	95 1-CHLOROHEXANE	18.372	17.273-19.471		41.00
		18.372	17.273-19.471		43.00
		18.372	17.273-19.471		55.00

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COMPOUND LISTING

Method file : /chem/VM/MSB5973.i/1b0809.b/M-18260B-m.m

Compound	RT	RT Window	RF	Mass
96 1 2-DIBROMO-3-CHLOROPROPAN	18.552	17.566-19.537	1.133e-01	75.00
	18.552	17.566-19.537		156.90
	18.552	17.566-19.537		154.90
97 5-METHYL-2-HEXANONE (MIAK)	18.722	17.623-19.821		43.00
	18.722	17.623-19.821		58.00
	18.722	17.623-19.821		57.00
98 1 2 4-TRICHLOROBENZENE	20.146	19.160-21.131	8.787e-01	179.90
	20.146	19.160-21.131		181.90
	20.146	19.160-21.131		145.00
99 trans-1,4-DICHLORO-2-BUTEN	20.446	19.065-21.827		53.00
	20.446	19.065-21.827		88.00
	20.446	19.065-21.827		89.00
100 HEXACHLOROBUTADIENE	20.541	19.556-21.527	4.492e-01	224.80
	20.541	19.556-21.527		222.80
	20.541	19.556-21.527		189.90
101 NAPHTHALENE	20.675	19.689-21.661	2.234e+00	128.00
	20.675	19.689-21.661		102.00
	20.675	19.689-21.661		51.00
102 alpha PINENE	20.750	19.651-21.849		93.00
	20.750	19.651-21.849		92.00
	20.750	19.651-21.849		91.00
103 beta PINENE	20.750	19.651-21.849		93.00
	20.750	19.651-21.849		92.00
	20.750	19.651-21.849		91.00
104 1 2 3-TRICHLOROBENZENE	21.241	20.496-21.986	7.900e-01	180.00
	21.241	20.496-21.986		182.00
105 PENTACHLOROETHANE	22.047	20.948-23.146		167.00
	22.047	20.948-23.146		130.00
106 2-methylnapthalene	30.000	29.500-30.500		142.00
	30.000	29.500-30.500		141.00
	30.000	29.500-30.500		0.00

SEMI-VOLATILE COMPOUNDS BY GC/MS (8270C)

Approved by:

R. Wayne Robbins   Jan 11, 2000

Title: STL-SL Technical Manager   Date

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1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of various semi-volatile organic compounds (SVOC) in groundwater, TCLP and SPLP leachates, soils, sediments, waste, and solid sample extracts. The attached quantitation report (Appendix B) lists the routine target compounds, the retention times of the target compounds, the characteristic ions of the target compounds, and the internal standard association of each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for the target compounds are given in Section 5 of the current revision of the STL Savannah Laboratories' *Laboratory Quality Plan.*

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the mass spectra determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 This procedure is based on the guidance provided in SW-846 Method 8270C.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

3.4 The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of the extract. The traps should be changed every three months and disposed of in accordance with STL-SL SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products such as diesel or waste oil.

4.3 Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days from TCLP leaching procedure	40 days

Refrigerator temperature acceptance criterion is less than 6C with no frozen samples.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph- Hewlett-Packard (HP) 5890 or equivalent with compatible autosampler, splitless injector, and direct capillary interface. The exit vent of the splitless injector must have a carbon trip in-line to collect the semivolatile compounds that are vented during the injection of extracts. The carbon traps should be changed every three months.

6.2 Mass spectrometer- HP5971, HP5972, HP5973 or equivalent

6.3 Recommended Capillary column-HP-5MS, 30m x 0.25mm ID x 0.25um film thickness or equivalent column.

6.4 Data system- compatible with GC/MS system

6.5 Microsyringes- appropriate volumes

6.6 Volumetric flasks, Class A-appropriate volumes

6.7 Autosampler vials and crimper, compatible with autosampler

7.0 REAGENTS

Reagents must be tracked in accordance with STL-SL SOP AN44: *Reagent Traceability.*

7.1 Methylene chloride-pesticide residue grade, for preparation of standards

7.2 Acetone-pesticide residue grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with STL-SL SOP AN41: *Standard Material Traceability*. General guidance on the preparation of standards is given in sTL-SL SOP AN43: *Standard Preparation.*

— The lab should purchase certified solutions from STL-SL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See STL-SL SOP AN43 for guidance for standard preparation from neat materials.

8.1 Preparation of the Stocks from Neat Standards

The steps for the preparation of primary stock standards from neat materials are given in STL-SL SOP *AN43: Standard Preparation*. The standards should be prepared in methylene chloride but may require other solvents to dissolve the material.

8.2 Preparation the calibration standards from the stock standards

A minimum of five calibration standards are prepared. The concentrations of the stock standards are in the 1000-10000ug/mL range. The recommended standards are listed in Section 10.2. The lowest level standard should be at the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve and seven calibration points are required for a polynomial regression curve (see SW-846 Method 8000B). Internal standards should be added to each standard to give a final concentration of 40 ug/mL.

Each lab should develop controlled recipes that can be posted or maintained in appropriate logbooks.

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP	Extraction Technique
Aqueous, TCLP leachates	EX30	Continuous Liquid-liquid Extraction
Aqueous, TCLP leachates	EX35	Separatory Funnel
Soils/Sediments	EX40	Sonication
Wastes	EX42	Waste dilution

9.2 The sample concentration procedures are given in STL-SL SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (GPC-STL-SL SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products.

10.0 PROCEDURE

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must optimize and document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:
HP-5MS 30m x 0.25mm ID x 0.25um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:
Initial column temperature: 45 C for 3 minutes
Column temperature program: 10C per minute
Final column temperature: 300C (until at least one minute past the elution time of Benzo (g,h,i) perylene).

GC injector parameters
Injector temperature: 250-270°C
Injection type: split, approximately 1:10 or splitless injection
Injector liner: 4mm ID quartz or 4mm glass, deactivated (single "Gooseneck")
Sample injection volume: 1-2uL

Mass Spectrometer and interface parameters
Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: 35-500amu, with a scan time of 1.0 scans per second or greater

10.2 Calibration

A minimum of five calibration standards are prepared and analyzed. The recommended TCL standards are 10, 50, 80, 100, 200, and 350ug/mL. The recommended Appendix IX concentrations are 10, 20, 50, 80, 100, 120, and 160ug/mL. The lowest level standard should be at or below the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve and seven calibration points are required for a polynomial regression curve (see SW-846 Method 8000B).

10.2.1 Fifty nanograms of DFTPP must be analyzed at the beginning of each 12-hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of DFTPP. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage.

10.2.1.1 Prepare a 50 ng/uL solution of tune/ column evaluation standard containing each of the following compounds at 50 ug/mL in methylene chloride: DFTPP, pentachlorophenol, p,p'-DDT, and benzidine.

10.2.1.2 Analyze a 1uL aliquot of the tune/column evaluation solution.

10.2.1.3 Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary (Appendix A). Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.2.1.4 Benzidine and pentachlorophenol should be present at their normal responses with no peak tailing visible. This is a good check on the system: if pentachlorophenol (a CCC) does not respond well, the calibration standard should not be analyzed. Perform injector port and column maintenance and reanalyze the tune/column evaluation standard.

The percent breakdown of p,p'- DDT is calculated using the following equation. The percent breakdown should not exceed 20%.

$$\%Breakdown = \frac{(areaDDE + areaDDD)}{(areaDDT + areaDDE + areaDDD)} x\,100$$

Areas from the total ion chromatogram are used to calculate DDT breakdown.

10.2.2 After the DFTPP criteria and column evaluation criteria have been met, the initial calibration standards are analyzed

10.2.2.1 Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range. See section 10.2 for guidance regarding calibration levels.

10.2.2.2 Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts.

10.2.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.2.4 Calculate the relative response factor for each compound as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured
(See the attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

10.2.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3 .... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

NOTE: As noted previously, some target compounds may have fewer than five calibration standard levels.

10.2.6 Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_1} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.2.7 Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\ 100$$

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10.2.8 Evaluation of the Initial Calibration

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in the SOP Summary (Appendix A). The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of sample extracts can begin.

If the CCC and SPCC criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards.

10.2.8 After the initial calibration criteria (CCC/SPCC) have been met, each target is evaluated for linearity.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated-with the correct internal standard)
Cx = concentration of the target compound being measured (ug/mL)
Cis = concentration of the internal standard (ug/mL)

A linear, quadratic, or higher order regression fit may be used to define the concentration/response relationship. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples. Regression curves may be forced through zero but it is recommended that the curve be evaluated without forcing through zero first and then with the curve forced through the origin. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire concentration range.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of the tune/column evaluation solution (10.2.1.1). The tune and column evaluation criteria (10.2.1.3 and 10.2.1.4) must be met before the analysis of the calibration check standards can take place.

10.3.1 After the tune and column evaluation criteria have been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a mid level concentration. The CCC and SPCC criteria (SOP Summary, Appendix A) must be met before the analysis of samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration (ug/mL)
Cccv = measured concentration using the selected quantitation method (ug/mL)

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If these criteria are not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.3.2 The continuing calibration verification standard (CCV) must also be evaluated for internal standard retention time and response.

If the retention time of any internal standard changes by more than 30 seconds from the last 12-hour calibration check, the analytical system must be inspected for problems and corrective action instituted.

If the extracted ion current profile (EICP) area for any of the internal standards in the CCV changes by more than a factor of two (-50% to +100%) from the last initial calibration sequence, the analytical system must be inspected for problems and corrective action instituted.

10.4 Samples are analyzed only after the DFTPP criteria, column evaluation criteria and the calibration verification criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the tune/column evaluation standard and a mid-level calibration standard.

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- Minimum of five cal levels	Mid point calibration verification Optional RL: Standard-low point on cal curve
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

10.4.1 Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.4.2 Add 20-uL of the internal standard mix (2000 ug/mL) to each 1.0 mL aliquot of the sample extract. The concentration of the internal standard in the extract is 40 ug/mL.

10.4.3 Mix the contents of the autosampler vial by inverting several times.

10.4.4 Analyze the samples using the same analytical conditions used for the initial and continuing calibration standard. Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

10.4.5 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract are diluted to final volume of 1.0mL, the dilution factor is 10. (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (2000ug/mL)-Vistd	DF
1000	0	1000	20	1
500	500	1000	10*	2
200	800	1000	16*	5
100	900	1000	18*	10
50	950	1000	19*	20
20	980	1000	20*	50

*assumes dilution of a 1.0mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 40ug/mL using 20ul of a 2000ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 40ug/mL. The following equation can be used to determine the volume of the 2000ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd(uL) = 20uL - \left(\frac{Vext}{Vdil} \otimes 20ul\right)$$

Vistd = volume of 2000ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

11.1.1 Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the daily calibration standard or a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to positively identify a compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{\textit{retention time of the target compound}}{\textit{retention time of the associated internal standard}}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. Ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification..

11.1.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.2.2 The relative intensities of the major ions should agree within +/-30%.

11.1.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.1.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.1.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.1.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult senior analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.1.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculations assume that the same volume is injected for standards and samples.

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{V} \otimes DF$$

where

Cis =	concentration of the internal standard (ug/mL)
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract (mL)
V =	volume of sample extract (L)
DF =	dilution factor

Soils

$$TIC\ (ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Cis =	concentration of the internal standard, ug/mL
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract mL
W =	weight of sample analyzed (kg)

solids = decimal equivalent of percent solids

11.2 Calculations for Samples-Internal Standard Technique

These calculations assume that the same volume is injected for standards and samples and that the standards and samples have the same concentration of internal standard.

11.2.1 Aqueous Samples

11.2.1.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.2 If a regression curve is used, the concentration is given:

$$concentration(ug/L) = Ccurve \otimes \frac{F}{V} \otimes DF$$

where

C_{curve} =	concentration from curve (ug/mL)
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.3 The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where

F =	final volume of extract (mL)
Fqap =	1.0mL
Vqap =	1.0L
V =	volume of sample extracted
DF =	dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

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11.2.2 Soils

11.2.2.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

11.2.2.2 If the regression curve is used, the concentration is given:

$$conc(ug/kg,dw) = Ccurve \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ccurve =	concentration from curve(ug/mL)
W =	weight of sample extracted (kg)
F =	final volume of extract (mL)
solids =	(percent solids)/100)
DF =	dilution factor

11.2.2.3 The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100

The SL CQAP assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching*. STL-SL SOP AN02 also describes the procedure for evaluating batch-specific QC. The QA/QC criteria are summarized in the SOP Summary (Appendix A).

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must participate in the analysis of samples by this procedure in accordance with STL-SL SOP CA92: *Evaluation of IDOCs.*

12.3 Method Detection Limit

The method detection limit is determined in accordance with STL-SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. See Section 10 of the current STL-SL Laboratory Quality Manual.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time.

15.0 REFERENCES

15.1 *STL Savannah Laboratories' Laboratory Quality Manual* current revisions.

15.2 Method 8270C:*Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

APPENDIX A
8270C SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification standard RL Standard (lowest point on calibration curve if required by client or state-specific QAP)
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)

m/e	Ion Abundance Criteria (1)
51	30-80% of mass 442
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	25-75% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5.0-9.0% of mass 198
275	10-30% of mass 198
365	Greater than 0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15.0-24.0% of mass 442

(1) 8270 criteria taken from CLP OLMO4.0 (January 1998). The use of alternate criteria is expressly allowed in SW-846 Method 8270C.

APPENDIX A
8270C SOP SUMMARY

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Phenol, 1,4-Dichlorobenzene, 2-Nitrophenol, 2,4-Dichlorophenol, Hexachlorobutadiene, 4-Chloro-3-methylphenol, 2,4,6-Trichlorophenol, Acenapthene, N-Nitrosodiphenylamine, Pentachlorophenol, Fluoranthene, Di-n-octylphthalate, Benzo(a) pyrene

System Performance Check Compounds-SPCC
N-Nitrosodi-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol, 4-Nitrophenol

Initial Calibration	Continuing Calibration*
CCC: <= 30% RSD	CCC: <= 20% difference from initial calibration
SPCC: RRFavg >= 0.050	SPCC: RRF>= 0.050

*If CCC and/or SPCC do not meet the stated criteria, all targets that are reported must meet the CCC criteria.

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

The results for all target compounds are evaluated for linearity. If the %RSD is less than 15%, the calibration is assumed linear through the origin and the average response factor can be used for quantitation. If the average response factor for the target exceeds 15% (including any CCC), the analyst must use the calibration curve option.

NOTE: The lab has the option of using a regression curve for all analytes.

A linear, quadratic, or higher order regression fit may be used to define the concentration/response relationship. If the correlation coefficient of the linear regression curve or the coefficient of determination of a higher order fit is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range. The minimum number of calibration standards required for a regression curve are given in the following table:

Type of curve	Minimum Number of Calibration Points
Linear (first order)	5
Quadratic (second order)	6
Polynomial(third)	7

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 50ng Pentachlorophenol - 50ng Benzidine – 50ng p,p'-DDT 50ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
		Pentachlorophenol and benzidine - present at usual response with no peak tailing visible p,p'-DDT - %breakdown <20%	-Reanalyze -Perform injector port maintenance and reanalyze -Cut more than usual length of column and reanalyze -Replace column
Initial Calibration	After Tune Check and when calibration verification standard fails acceptance criteria. All initial calibration standards	CCC: %RSD < 30% SPCC: RRFavg > 0.050 Use regression curve for quantitation if %RSD for any target compound exceeds 15%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples	CCC: %Difference <= 20% Or %Drift <= 20% SPCC: RRF >= 0.050	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence Areas in samples should be evaluated for gross error . Consult supervisor Retention time of internal standard must be +/-30 seconds from internal standard in previous CCV.	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available -Recalibrate

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QC Item	Frequency	Acceptance Criteria	Corrective Action
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within STL-SL LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in LQM	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Lab Control Standard (LCS) - QAP subset	Per batch See STL SOP AN02	Within STL-SL LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See AN02	Within STL-SL LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
RL Standard (reporting limit)	Daily (optional)-lowest point on calibration curve if required by client or state-specific QAP	Detected at reasonable sensitivity	-Evaluate integrations and spectra; -Reanalyze -Prepare new standard and reanalyze
Initial Demonstration of Capability (IDOC)	Each work group	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	Annually for each routine matrix See STL SL SOP CA90	Evaluate according to STL-SL SOP CA90	Evaluate according to STL-SL SOP CA90

APPENDIX B- EXAMPLE QUANTITATION REPORT

Savannah Laboratories

Semivolatile REPORT SW-846 Method 8270C

Data file : /chem/SM/MSE5973.i/1e0107.b/eq612.d
Lab Smp Id: SMTCL-200 Client Smp ID: SMTCL-200
Inj Date : 07-JAN-2000 08:56
Operator : Inst ID: MSE5973.i
Smp Info : 1/7/00
Misc Info : SMTCL-200 SMC03-7-1
Comment :
Method : /chem/SM/MSE5973.i/1e0107.b/e-b8270C-m.m
Meth Date : 07-Jan-2000 10:19 jstingl Quant Type: ISTD
Cal Date : 28-DEC-1999 19:45 Cal File: eq582.d
Als bottle: 2 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: t199{350}.sub
Target Version: 3.40
Processing Host: cserver1

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (NG)	ON-COL (NG)
*	1 1,4-Dichlorobenzene-d4	152	5.109	5.109	(1.000)	138890		
	2 1,4-Dioxane	88	2.284	2.284	(0.447)	365537	200.000	180
	3 Pyridine	79	2.573	2.573	(0.504)	990777	200.000	170
	4 N-Nitrosodimethylamine	42	2.563	2.563	(0.502)	372916	200.000	180
$	5 2-Fluorophenol	112	3.784	3.784	(0.741)	783911	200.000	180
$	6 Phenol-d5	99	4.768	4.768	(0.933)	1100575	200.000	180
	7 Aniline	93	4.788	4.788	(0.937)	1279048	200.000	170
	8 Phenol	94	4.778	4.778	(0.935)	1157650	200.000	180
	9 Bis(2-chloroethyl)ether	63	4.861	4.861	(0.951)	629821	200.000	180
	10 2-Chlorophenol	128	4.912	4.912	(0.962)	921621	200.000	190
	11 1,3-Dichlorobenzene	146	5.068	5.068	(0.992)	1002450	200.000	190
	12 1,4-Dichlorobenzene	146	5.120	5.120	(1.002)	1011714	200.000	190
	13 Benzyl Alcohol	108	5.295	5.295	(1.036)	621327	200.000	180
	14 1,2-Dichlorobenzene	146	5.337	5.337	(1.045)	943597	200.000	190
	15 2-Methylphenol	107	5.440	5.440	(1.065)	742922	200.000	190
	16 bis(2-Chloroisopropyl)ether	45	5.471	5.471	(1.071)	1127568	200.000	190
	17 N-Nitroso-di-n-propylamine	70	5.637	5.637	(1.103)	652256	200.000	180
	18 3&4-Methylphenol	107	5.606	5.606	(1.097)	1071208	200.000	180
	19 Hexachloroethane	117	5.678	5.678	(1.111)	369084	200.000	190
*	20 Naphthalene-d8	136	6.620	6.620	(1.000)	576109		
$	21 Nitrobenzene-d5	82	5.772	5.772	(0.872)	999920	200.000	210
	22 Nitrobenzene	77	5.792	5.792	(0.875)	964588	200.000	200
	23 Isophorone	82	6.082	6.082	(0.919)	1688698	200.000	180
	24 2-Nitrophenol	139	6.175	6.175	(0.933)	524138	200.000	230
	25 2,4-Dimethyphenol	122	6.237	6.237	(0.942)	774066	200.000	180
	26 Bis(2-chloroethoxy)methane	93	6.362	6.362	(0.961)	1061177	200.000	180

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (NG)	ON-COL (NG)
	========================	====	==	======	======	========	=======	=======
	27 Benzoic acid	122	6.434	6.434	(0.972)	535495	200.000	180
	28 2,4-Dichlorophenol	162	6.475	6.475	(0.978)	777505	200.000	200
	29 1,2,4-Trichlorobenzene	180	6.569	6.569	(0.992)	813165	200.000	200
	30 Naphthalene	128	6.651	6.651	(1.005)	2635688	200.000	220
	31 4-Chloroaniline	127	6.755	6.755	(1.020)	1098581	200.000	180
	32 Hexachlorobutadiene	225	6.889	6.889	(1.041)	426857	200.000	200
	33 4-Chloro-3-methylphenol	107	7.417	7.417	(1.120)	827216	200.000	190
	34 2-Methylnaphthalene	142	7.572	7.572	(1.144)	1802569	200.000	190
	35 1-Methylnaphthalene	142	7.717	7.717	(1.166)	1730785	200.000	190
*	36 Acenaphthene-d10	164	9.094	9.094	(1.000)	329747		
	37 Hexachlorocyclopentadiene	237	7.893	7.893	(0.868)	529072	200.000	230
	38 2,4,6-Trichlorophenol	196	8.018	8.018	(0.882)	561125	200.000	200
	39 2,4,5-Trichlorophenol	196	8.069	8.069	(0.887)	595405	200.000	200
$	40 2-Fluorobiphenyl	172	8.131	8.131	(0.894)	1881161	200.000	190
	41 2-Chloronaphthalene	162	8.256	8.256	(0.908)	1714878	200.000	190
	42 2-Nitroaniline	65	8.463	8.463	(0.931)	551375	200.000	210
	43 Dimethylphthalate	163	8.804	8.804	(0.968)	1947578	200.000	180
	44 2,6-Dinitrotoluene	165	8.897	8.897	(0.978)	459158	200.000	230
	45 Acenaphthylene	152	8.866	8.866	(0.975)	2701962	200.000	180
	46 3-Nitroaniline	138	9.094	9.094	(1.000)	549348	200.000	210
	47 Acenaphthene	154	9.146	9.146	(1.006)	1648463	200.000	190
	48 2,4-Dinitrophenol	184	9.239	9.239	(1.016)	265203	200.000	300
	49 4-Nitrophenol	65	9.373	9.373	(1.031)	383510	200.000	200
	50 Dibenzofuran	168	9.384	9.384	(1.032)	2380681	200.000	180
	51 2,4-Dinitrotoluene	165	9.477	9.477	(1.042)	643843	200.000	210
	52 2,3,4,5-Tetrachlorophenol	232	9.632	9.632	(1.059)	557848	200.000	220
	53 2,3,4,6-Tetrachlorophenol	232	9.653	9.653	(1.061)	528217	200.000	200
	54 Diethylphthalate	149	9.891	9.891	(1.088)	1910197	200.000	180
	55 Fluorene	166	9.912	9.912	(1.090)	1962664	200.000	230
	56 4-Chlorophenyl-phenylether	204	9.943	9.943	(1.093)	906483	200.000	200
	57 4-Nitroaniline	138	10.046	10.046	(1.105)	572192	200.000	200
$	58 2,4,6-Tribromophenol	330	10.315	10.315	(1.134)	303242	200.000	230
*	59 Phenanthrene-d10	188	11.350	11.350	(1.000)	621681		
	60 4,6-Dinitro-2-methylphenol	198	10.098	10.098	(0.890)	369634	200.000	300
	61 N-Nitrosodiphenylamine	169	10.139	10.139	(0.893)	1441356	200.000	180
	62 1,2-Diphenylhydrazine	77	10.181	10.181	(0.897)	2095566	200.000	180
	63 4-Bromophenyl-phenylether	248	10.688	10.688	(0.942)	539491	200.000	190
	64 Hexachlorobenzene	284	10.895	10.895	(0.960)	581783	200.000	200
	65 Pentachlorophenol	266	11.185	11.185	(0.985)	364412	200.000	200
	66 Phenanthrene	178	11.392	11.392	(1.004)	2871313	200.000	220
	67 Anthracene	178	11.464	11.464	(1.010)	2895262	200.000	220
	68 Carbazole	167	11.754	11.754	(1.036)	2838733	200.000	190
	69 Di-n-Butylphthalate	149	12.458	12.458	(1.098)	3186408	200.000	200
	70 Fluoranthene	202	13.286	13.286	(1.171)	2950951	200.000	240
	71 Benzidine	184	13.534	13.534	(0.887)	1713183	200.000	210
*	72 Chrysene-d12	240	15.263	15.263	(1.000)	357835		
	73 Pyrene	202	13.607	13.607	(0.891)	2729353	200.000	200

							AMOUNTS	
	QUANT SIG						CAL-AMT	ON-COL
Compounds	MASS	RT	EXP RT	REL RT	RESPONSE		(NG)	(NG)
$ 74 Terphenyl-d14	244	13.886	13.886	(0.910)	1726251		200.000	190
75 Butylbenzylphthalate	149	14.621	14.621	(0.958)	1010594		200.000	170
76 3,3'-Dichlorobenzidine	252	15.263	15.263	(1.000)	783655		200.000	210
77 Benzo(a)Anthracene	228	15.242	15.242	(0.999)	1960776		200.000	190
78 Bis(2-ethylhexyl)phthalate	149	15.490	15.490	(1.015)	1351209		200.000	160
79 Chrysene	228	15.304	15.304	(1.003)	1797418		200.000	180
* 80 Perylene-d12	264	17.229	17.229	(1.000)	416323			
81 Di-n-octylphthalate	149	16.349	16.349	(1.071)	2333973		200.000	180
82 Benzo(b)fluoranthene	252	16.743	16.743	(0.972)	2436436		200.000	210
83 Benzo(k)fluoranthene	252	16.784	16.784	(0.974)	1959383		200.000	230
84 Benzo(a)pyrene	252	17.167	17.167	(0.996)	2003135		200.000	210
85 Indeno(1,2,3-cd)pyrene	276	18.689	18.689	(1.224)	2303273		200.000	180
86 Dibenzo(a,h)anthracene	278	18.730	18.730	(1.087)	1854734		200.000	190
87 Benzo(g,h,i)perylene	276	19.092	19.092	(1.108)	1923577		200.000	190

ORGANOCHLORINE PESTICIDES AND PCBs BY GC
(Methods 608 and 8081A/8082)

Approved by:

Jan 7, 2000

Title: STL-SL Technical Manager   Date

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1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of various chlorinated pesticides and polychlorinated biphenyls (PCBs) in liquid and soil extracts. Appendix A gives an example of the retention times of the routine target compounds. A summary of the method QC requirements is provided in the Appendix C.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria are given in Section 5 of the current revision of the STL Savannah Laboratories Laboratory Quality Manual.

The procedures for chlorinated pesticides (8081A) and PCBs (8082) are given as separate methods separated in the latest update of SW-846. Until this update, the pesticides and PCBs were combined in both SW-846 Methods 8080 and 8081. The extraction and the analysis are combined in this SOP 1) to reduce the time of extraction and analysis; and 2) to reduce the amount of solvent used in the procedures (one extraction instead of two). If interferences or high levels of non-PCB compounds are present, a portion of the extract will be subjected to the acid cleanup and reanalyzed. Note that if the list of target analytes includes only a limited list of components (i.e. Toxaphene, Chlordane, or PCBs), these procedures may be abbreviated to address only the analytes of interest.

2.0 SUMMARY OF METHOD

2.1 Aqueous and leachate samples are extracted with methylene chloride using a continuous liquid/liquid extractor (SOP EX30) or separatory funnel (SOP EX35). Soils are extracted with 1:1 hexane/acetone or 1:1 acetone/methylene chloride using a sonic dismembrator (SOP EX40). The solvent is evaporated, the residue exchanged into hexane, and the sample adjusted to a final volume of 10mL or less. The preparation may also incorporate Florisil, copper (sulfur), acid (PCBs and Toxaphene only), or gel permeation chromatography (GPC) cleanups. Analysis of the extract is routinely performed on a GC equipped with dual capillary columns (different phases) connected to dual electron capture (EC) detectors, allowing simultaneous detection and confirmation of the target compounds. GC/MS confirmation can also be employed if analyte concentration is sufficiently high or if the sample extract is concentrated to an appropriate final volume. Quantitation may be performed using the external or internal standards calibration technique. The procedures for waste dilution are given in STL-SL SOP EX42.

2.2 This method is based on the guidance in SW-846 Methods 8000B, 8081A, and 8082 and 40 CFR 136 Method 608.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially hazardous situations.

3.2 The analyst should wear an apron or lab coat, gloves, and eye protection when handling extracts. Dilutions should be performed under a hood or in a well-ventilated area.

3.3 The analyst must be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in the analysis of pesticides and PCBs. Many of these compounds are suspected carcinogens.

4.0 INTERFERENCES

4.1 Glassware should be scrupulously cleaned and solvent-rinsed in accordance with STL-SL SOP AN60 to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination form the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. See Section 9 for a table summary of the cleanups that may be employed to eliminate or reduce interferences. If matrix interferences continue after a clean up has been performed, the sample is diluted as needed for data analysis. If a cleanup is used, the method blank must also be subjected to the cleanup.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

Aqueous samples are collected in 1-L glass containers with Teflon-lined caps. Soil/sediment samples are collected in wide mouth glass jars equipped with Teflon lined caps. No preservative is added. The samples are iced at the time of collection and maintained at 4°C (less than 6C with no frozen samples) until extraction. Extraction must be performed within 7 days for aqueous samples and within 14 days of sampling for soils/solids. The extracts must be stored at 4°C (less than 6C) and must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperatures programmable, equipped with single or dual electron capture (EC) detectors and a compatible autosampler. The GC should be capable of housing two columns and have make-up gas lines for each detector

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended

DB-5 fused silica capillary column 30 M x 0.53 mm ID x 1.5 um film (J&W or equivalent)
DB-608 fused silica capillary column 30 M x 0.53 mm ID x 0.83 um film (J&W or equivalent)
<Or>
DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-17 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
<Or>
DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-1701 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)

6.4 Microsyringes, appropriate volumes

6.5 Volumetric flasks, Class A, appropriate volumes

6.6 Autosampler vials, septa, and caps; compatible with the autosampler

7.0 REAGENTS

Hexane- pesticide grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with STL-SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in STL - SL SOP AN43:*Standard Preparation.*

The lab should purchase certified solutions from STL-SL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See STL-SL SOP AN43 for guidance for standard preparation.

Calibration Standard Recipes

The standard concentrations given in Appendix B are provided for guidance. The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. Note that the preparation of some of the calibration standards will include intermediate level standards.

The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the STL-SL LQM or client QAPP. The remaining standards will define the working range of the analytical system. When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

9.0 SAMPLE PREPARATION

The sample preparation and cleanup procedures are described in the following SOPs:

PROCEDURE	MATRIX	STL-SL SOP
Continuous Liquid-liquid extraction	aqueous and leachates	EX30
Separatory funnel extraction	aqueous and leachates	EX35
Ultrasonic extraction	soils and sediments	EX40
Waste dilution	Waste samples (oils, products, etc)	EX42
Zymark extract concentration	Aqueous, leachate, and soil extracts	EX50

CLEAN-UP PROCEDURE	STL-SL SOP	APPLICATION	EFFECTIVENESS
Florisil	EX62	pest/PCBs	Eliminates polar non-target compounds
Sulfuric acid / permanganate	EX60	PCBs and Toxaphene	Eliminates some unsaturated hydrocarbon interferences
Copper	EX60	pest/PCBs	Eliminates elemental sulfur
GPC	EX61	pest/PCBs	Eliminates high molecular weight non-target compounds and sulfur

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions

The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations may be used for the analysis of pesticides and PCBs. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide simultaneous detection and confirmation of the target analytes. The following stationary phase pairs are recommended:

DB-5 fused silica capillary column 30 M x 0.53 mm ID x 1.5 um film (J&W or equivalent)
DB-608 fused silica capillary column 30 M x 0.53 mm ID x 0.83 um film (J&W or equivalent)

DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-17 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)

DB-5 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)
DB-1701 fused silica capillary column 30 M x 0.32 mm ID x 0.5 um film (J&W or equivalent)

A guard column is recommended to help protect the analytical columns.

10.1.2 Example GC Parameters

Injector: 220 – 240°C
Detector: 300 – 320 °C
Carrier Gas Flow: Helium at 5 mL/min (per column)
Make-up Gas Flow: Nitrogen at 25 mL/min (per detector)-see manufacturer's recommended flows

Example chromatogram temperature program:

Initial Temp:	160 C
Initial Hold:	4.0 min
Program Rate:	10 C/min
Final Temp:	270 C (hold for 10 minutes)
Injected Volume:	2-4uL - 1-2uL per column (single injection into guard column and "Y" splitter)

NOTE: These conditions and parameters are given for guidance. The conditions and parameters may be modified to optimize the analytical system.

10.2 Column Evaluation

The column(s) must be evaluated prior to the analysis of the calibration standards. The column evaluation is performed by injecting Endrin and p,p'-DDT and calculating the percent breakdown of these compounds.. The column evaluation does not have to be performed if PCBs only are the target compounds. PCBs are stable and not subject to breakdown in the injection port.

If the instrument has not been in use for more than one day, a "priming" analysis may be beneficial. The analysis of a relatively high concentration pesticide or PCB standard may help to stabilize the response of the very sensitive EC detector. Inject a standard that is about 10x the concentration of the highest calibration standard and allow the instrument to cycle through the temperature program. It is not necessary to acquire the data but the baseline should be monitored before and after the priming analysis to gauge the condition of the detector. A hexane blank should be analyzed after the analysis of the priming standard and before the %breakdown check.

NOTE: The "priming" standard should be injected manually to avoid contaminating the autosampler syringe.

Inject the Endrin/DDT breakdown standard. Check the peak integrations and calculate the breakdown as follows:

$$\%\text{Breakdown Endrin} = \frac{\text{Area(Endrin Aldehyde} + \text{Endrin Ketone)}}{\text{Area(Endrin} + \text{Endrin aldehyde} + \text{Endrin Ketone)}} \otimes 100$$

$$\%\text{Breakdown DDT} = \frac{\text{Area(DDE} + \text{DDD)}}{\text{Area(DDT} + \text{DDE} + \text{DDD)}} \otimes 100$$

The breakdown for each compound must be less than 15%. If the breakdown exceeds 15%, the instrument will require column and/or injector port maintenance. The maintenance may include but is not limited to replacing the septum, clipping the front of the column, and replacing the glass injector sleeve.

10.2 Initial Calibration

The external standard calibration technique is routinely employed for the determination of the concentration of pesticides and PCBs. The lab also has the option of using internal standard calibration. Pentachloronitrobenzene (PCNB) or 2-Nitro-1-bromobenzeneis are suggested for use as internal standards.

10.2.1 Prepare and analyze the calibration standards. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in the SOP Summary.

Note that the following offers two (2) options for calibration and quantitation – average CF or regression curve. Only one need be chosen per analyte.

When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

10.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?
>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?
>Are the peaks symmetrical? Is there tailing or fronting?
>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.3 Average CF or RF Option:
Calculate the calibration factor (CF) or the response factor (RF) of each calibration standard, the average calibration or response factor and the relative standard deviation using the following equations:

Calibration factor-external standard

$$CF = \frac{Rt}{Ct}$$

where
Rt = response (area or height) of the target compound
Ct = concentration of the target compound, usually expressed as ug/mL

Response factor-internal standard

$$RF = \frac{Ac}{Ais} \otimes \frac{Cis}{Cc}$$

where

Ac = area of the target compound
Ais = area of the internal standard
Cc = concentration or mass on-column of the target compound (ug/mL)
Cis = concentration or mass on-column of the internal standard (ug/mL)

Average Calibration Factor or Response Factor

$$CF_{avg} = \frac{CF_1 + CF_2 + CF_3 + \ldots CFn}{n}$$

Relative Standard Deviation

$$\% RSD = \frac{standard\ deviation}{CF_{avg}} \otimes 100$$

$$Standard\ \ Deviation = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF_{avg}})^2}{n-1}}$$

Where

CF_i = calibration factor (or response factor) of the individual calibration level
CF_{avg}= average calibration factor (or response factor)

10.2.4 Initial Calibration Criteria:

600-series: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear and the average calibration factor may be used for quantitation.

8000-series: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear and the average calibration factor may be used for quantitation.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average calibration factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

10.2.5 Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples. For 8000-series methods, a minimum of five points is required for a linear regression, six points for a second order curve, and seven or more for higher order fits. It is recommended that only linear and quadratic (second order) curves be used for quantitation. See STL-STL SL SOP AN67 for guidance on evaluation of calibration curves.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

10.3 Calibration Verification

Calibration is verified at the frequency given in the SOP Summary. Note that the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive for the compound(s) failing high are detected, these samples may be reported.

10.3.1 Analyze the mid-level standard(s). Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration:

$$PercentDrift = \left|\frac{Cccv - Ctrue}{Ctrue}\right| \otimes 100$$

where
Cccv = concentration of CCV (ug/mL) quanted from regression curve or CFavg or RFavg
Ctrue = true concentration of the CCV (ug/mL)

$$PercentDifference = \left|\frac{CFccv - CFavg}{CFavg}\right| \otimes 100$$

where
CFccv = calibration factor (or RF) of CCV
CFavg = average calibration factor (or RFavg) from initial calibration

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10.3.2 Continuing Calibration Verification Criteria

Response Criteria:
If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

600-series: If the percent drift or percent difference is less than or equal to 15%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

8000-series: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor is used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average calibration factor or regression curve from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

All samples analyzed must be bracketed by acceptable CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Retention Time Criteria
The retention time for the CCV must fall within the daily retention time window as defined in STL-SL SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses.*

10.4 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the SOP Summary in Appendix C.

10.4.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.4.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted in hexane and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve.

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve (i.e., a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client). Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in STL-SL SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses*. If internal standard calibration is used, the determination of absolute retention time windows is not required. Relative retention times, as described in Section 11.1.4, are used to identify the target compounds.

11.0 DATA ANALYSIS/CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times), the recovery and retention time shift of the surrogate compounds. whether the peak response falls within the working range of the calibration, and the integration of the peaks. Manual integration must be documented in accordance with STL-SL SOP AN65. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data.

The judgement and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?

11.1 Qualitative analysis

Identification of the surrogates and target compounds is based on retention time. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peaks areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks, which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (10.4.2).

11.1.1 The surrogates should be evaluated first to check for shifts in retention times and to evaluate the surrogate recovery. The recovery criteria are given in Section 5 of the STL-Laboratory Quality Manual (LQM).

A minimum of two surrogates are spiked into each sample and QC item prior to preparation. 2,3,4,6-tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are the recommended surrogates. DCB should be evaluated as the primary surrogate; TCMX is evaluated if there is matrix interference with DCB.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Generally, it is expedient to calculate the recovery of both surrogates on one chromatogram. If these recoveries are deemed acceptable, the other channel is not evaluated for recovery. If interferences are suspected, evaluate recoveries on the other channel. Note that given this nature, an extract is considered acceptable if one of the 4 potentially calculated recoveries is acceptable.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to section 13 of the current Laboratory Quality Manual regarding this issue,

11.1.2 Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

If the concentration is below the detection limit, the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.3.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds.

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

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If the relative percent difference is less than 40%, the presence of the target compound is confirmed and the higher concentration is reported. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A large relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. If interference is detected, flag the result to note the disparity between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

The following table summarizes the general guidance for the evaluating of chromatographic data.

PEAK INFORMATION	ACTION	REPORT(1)
No peaks found on primary or secondary column		Report < RL
Peak found within RTW on primary column	Peak is tentatively identified as the target. If, in the professional judgement of the analyst and supervisor, the peak within the RTW can be reasonably excluded as a target, the result may be reported as a "non detect".	If concentration < RL, report < RL If concentration > RL, evaluate confirmation column.
Peak found within RTW on confirmation column	Peak is tentatively confirmed as the target. If, in the professional judgement of the analyst and supervisor, the peak within the RTW can be reasonably excluded as a target, the result may be reported as a "non-detect".	If concentration < RL, report <RL If concentration >RL, calculate %RPD -if %RPD <=40, report highest concentration of primary and confirmation analyses -if %RPD >40, report the lowest result or the result that is most reasonable for the sample matrix. and flag result to note the disparity Case narrative or note to PM may be required for complex matrices.

(1)RL may be the STL-SL Reporting Limit in Table 5 of the LQM or may be defined by the client QAP or contract.

The analyst must clearly show how the reported sample results were determined. Manual calculations and integrations must be documented as described in STL SOP AN65.

11.1.4 Identification "Tools"

Analysis by GC/MS (scan or SIM) may be used to confirm the presence of the target compounds (see STL-SL SOP SM06: *Guidelines for SIM Analysis by GC/MS.)*

11.1.4.1 Relative Retention Time

The retention time of a surrogate compound provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification. Another useful tool that employs a similar idea to the RRT is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.1.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

11.1.5 Qualitative Analysis of Multiple Peak Compounds

Identification of multicomponent pesticides/PCBs is based on the recognition of their chromatographic patterns. Quantitation is performed using the area of characteristic peaks in the sample and standard using external calibration procedures.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be warranted. Suggested cleanup options are given in Section 4.

11.1.5.1 PCBs as Aroclors

PCBs are generally reported as Aroclors. The Aroclors have varying levels of PCB congeners with the last two numbers in the Aroclor designation indicating the weight percent of chlorine. For example, AR1221 is 21% chlorine by weight; AR1260 is 60% chlorine by weight. The 12- in the Aroclor designation represents the biphenyl molecule. The exception to this naming convention is AR1016, which is about 42% chlorine by weight. (Note that AR1026 and AR1242 have similar chromatograms - both Aroclors have almost the same weight of chlorine by weight and nearly the same PCB congeners.)

Aroclors are identified by matching the pattern of the sample with standards analyzed under the same analytical conditions. Interference may occur due to the presence of non-target analytes or due to "weathering" of the Aroclor in the environment. The presence of multiple Aroclors will also complicate the identification and quantitation of the Aroclors. Many matrix interferences may be reduced or eliminated by treating the sample extract with copper, sulfuric acid, and permanganate prior to analysis. STL SL SOP EX60 details this procedure.

NOTE: Do not use the acid or permanganate cleanup on the entire extract if pesticides are also to be reported as many of the pesticides are not stable in acid or strong oxidizer.

When a pattern matching an Aroclor is encountered, it may be quantitated using either the 3-5 characteristic peaks (recommended) or total area response. Total area quantitation should only be used as detailed below. Residues of either AR1016 or AR1260 are quantitated using the average RF/CF determined during initial calibration. The other Aroclors are quantitated against the RF/CF determined from their single-point analysis during initial calibration. Samples should be diluted when the amount of PCB in a sample extract exceeds the calibration range defined in initial calibration. Note that the AR1660 standard defines the working range for all the Aroclors (i.e. if AR1660 was calibrated from 0.10ug/mL to 5.0ug/ml, and a sample extract was analyzed containing 10ug/ml of AR1232, that extract would require dilution to get the amount of AR1232 to be less than 5.0ug/ml.)

In the 3-5 peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of PCB in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 3-5 resulting concentrations are averaged to provide the final result for the sample.

"Weathering" is the loss of part of the Aroclor pattern due to biological or chemical degradation of individual PCBs. When weathering is suspected, try to match the later eluting peaks first. Flag the results for a weathered Aroclor pattern as tentatively identified and make a note in the case narrative if provided.

The presence of multiple Aroclors can be a problem to identify since most Aroclors have at least a few PCBs in common. The easiest case would be to have early and late eluting Aroclors present. The most difficult cases will involve the presence of Aroclors with the same relative chlorine level. In cases where the identification of Aroclors cannot be clearly determined, quantify the Aroclors against the Aroclor that most closely matches the pattern of the sample using the total area of all peaks within the pattern range. The result should be flagged and noted in the case narrative if provided.

NOTE: When choosing individual peaks for quantitation, compare their responses in the sample and standard. If the peaks chosen do not correlate well (i.e. ratios to other peaks are close) between the sample and standard, review the chromatograms for other possible peaks for quantitation.

11.1.5.2 Toxaphene

Toxaphene is a mixture of chlorinated camphenes, which has a complex and characteristic pattern when analyzed by GC-ECD. A single Toxaphene standard is analyzed during the initial calibration for the purpose of pattern identification in samples. When a Toxaphene residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for Toxaphene should be analyzed. Alternatively, single points may be prepared with Toxaphene concentrations within 2x the Toxaphene quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the Toxaphene standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of Toxaphene-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours.

If the sample and standard chromatograms agree well, Toxaphene is quantitated using 5 characteristic peaks (similar to the PCB approach, above). If the sample and standard pattern do not agree as well (i.e. individual peak ratios do not agree as well, but all the major Toxaphene components in the standard are present in the sample), a total area integration is more appropriate. To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene components in the standard. Note that in order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample and that extraneous peaks or humps (contributed by non-Toxaphene components) are NOT included in the quantitation.

When Toxaphene is determined using the 5 peak approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionately larger or smaller in the sample compared to the standard.

In the 5-peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 5 resulting concentrations are averaged to provide the final result for the sample.

11.1.5.3 Technical Chlordane –

Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The following components are significant: α and γ Chlordane, trans-Nonachlor, Heptachlor, and Heptachlor-epoxide. The α and γ Chlordane isomers are the most prevalent and their detection as single components is a good indicator that Technical Chlordane may be present.

The following sections discuss three specific options: reporting Technical Chlordane, reporting Chlordane (not otherwise specified-"NOS"), and reporting the individual Chlordane components that can be identified under their individual CAS numbers.

When the GC pattern of the residue resembles that of the Technical Chlordane standard, quantitate Chlordane residues by comparing the area of 3 to 5 major peaks. Heptachlor and heptachlor epoxide should not be included in this quantitation but rather should be quantitated and reported separately.

The GC pattern of a Chlordane residue in a sample may differ considerably from that of the Technical Chlordane standard. In such instances, it may not be practical to relate a sample chromatogram back to the lab's Technical Chlordane Standard. Therefore, depending on the objectives of the analysis, the analyst may choose to report the sum of all the identifiable Chlordane components as "Chlordane (n.o.s.)" under the CAS number 57-74-9. This option should only be used at the direction of or in consultation with a project manager.

The third option is to quantitate the peaks of a-Chlordane and y-Chlordane separately against the appropriate reference materials, and report these individual components.. This option should be used only after consultation with the project manager. Note that quantitation values determined from the individual chlordane isomers will be substantially lower than those reported for the technical product.

When a Technical Chlordane residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for technical chlordane should be analyzed. Alternatively, single points may be prepared with technical chlordane concentrations within 2x the Technical Chlordane quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the technical chlordane standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of technical chlordane-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours. Note that these procedures are not necessary if the lab is reporting chlordane as the α and γ chlordane isomers, not as the technical product.

11.2 Calculations-External Standard

Aqueous/Liquid Samples

If the regression curve option is used, the sample concentration is calculated:

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{V}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

If the calibration factor option is chosen for quantitation:

$$Concentration(ug/L) = \frac{response}{CF_{avg}} \otimes \frac{F \otimes DF}{V}$$

where
Response = area (or height) of the target
CF_{avg} = average calibration factor (ICAL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

Soils/Solids Samples

Regression Curve

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
W = weight of sample extracted (kg)
solids = (percent solids)/100

If the calibration factor option is chosen for quantitation:

$$Concentration(ug/kg,dw) = \frac{response}{CF_{avg}} \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
response = area (or height) of the target
CF_{avg} = average calibration factor
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)
W = weight of sample extracted (kg)
solids = (percent solids)/100

11.3 Internal Standard Calibration

Aqueous Samples

$$Concentration(ug/L) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{V} \otimes DF$$

where

Ac = area of the target compound
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
V = volume of sample extracted (l)
DF =dilution factor

Soil Samples

$$Concentration(ug/kg,dw) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{W \otimes solids} \otimes DF$$

where

Ac = area of the target compounds
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
W =weight of sample extracted (kg)
Solids = (percent solids)/100
DF =dilution factor

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The **analytical batch** is discussed in STL-SL SOP AN02: *Analytical Batching*, and these criteria are summarized in the SOP Summary included in Appendix C. Calculation of QC data is also given in AN02.

12.2 The **method detection limit (MDL)** must be determined annually in each matrix of concern in accordance with STL-SL SOP CA90: *Procedure for Determination of Method Detection Limit (MDL).*

12.3 Each analyst must participate (individually or as part of a work group) in the analysis and evaluation of QC samples to demonstrate minimum proficiency in this procedure. The IDOC samples are processed in the same manner as routine samples and evaluated according to STL-SL SOP CA92.

13.0 PREVENTIVE MAINTENANCE

No items are included in this revision. See Section 10 of the current STL-SL LQM.

14.0 TROUBLESHOOTING

No items are included in this revision.

15.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

2. *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office: Washington, DC, July 1, 1988.

3. STL Savannah Laboratories' *Laboratory Quality Manual*, current revision.

Appendix A

TABLE 1		
Compound	RT COL 1	RT COL 2
Tetrachloro-m-xylene	5.26	6.65
Alpha-BHC	7.39	7.71
Gamma-BHC (Lindane)	8.63	8.65
Beta-BHC	8.80	8.39
Heptachlor	9.54	10.61
Delta-BHC	9.83	9.19
Aldrin	10.38	11.53
Heptachlor epoxide	11.96	12.51
Gamma-Chlordane	12.31	13.04
Alpha-Chlordane	12.72	13.41
Endosulfan I	12.86	13.42
4,4'-DDE	13.39	13.84
Dieldrin	13.63	14.05
Endrin	14.45	14.53
4,4'-DDD	14.75	14.83
Endosulfan I	14.97	14.78
4,4'-DDT	15.54	15.78
Endrin aldehyde	15.76	15.21
Endosulfan sulfate	15.98	15.71
Dibutyl chlorendate	16.73	17.80
Methoxylchlor	17.60	17.04
Endrin ketone	17.79	16.82
Decachlorobiphenyl	21.36	22.36
Technical chlordane	MR	MR
Toxaphene	MR	MR
AR 1221	MR	MR
AR1232	MR	MR
AR1016	MR	MR
AR1242	MR	MR
AR1248	MR	MR
AR1254	MR	MR
AR1260	MR	MR
Isodrin	11.85	12.45
Chlorobenzilate	14.51	14.79
AR1268	MR	MR

MR = multi-peak/multi-response compounds COL1 = DB608 COL2 = DB-5

Appendix B:CHLORINATED PESTICIDES AND PCBs STANDARDS

Routine Targets

Target compound	ISMA-1 (ug/mL)	ISMA-2 (ug/mL)	ISMA-3 (ug/mL)	ISMA-4 (ug/mL)	ISMA-5 (ug/mL)
TCMX, DCB (surr)	0.0025	0.0050	0.010	0.020	0.040
g-BHC(Lindane), Heptachlor, Heptachlor epoxide, Endosulfan I	0.0050	0.010	0.020	0.030	0.050
Dieldrin, p,p'-DDT, Endosulfan II, Endrin aldehyde, Methoxychlor	0.010	0.020	0.040	0.060	0.10

Target compound	ISMB-1 (ug/mL)	ISMB-2 (ug/mL)	ISMB-3 (ug/mL)	ISMB-4 (ug/mL)	ISMB-5 (ug/mL)
TCMX,DCB (surr)	0.0025	0.0050	0.010	0.020	0.040
a-BHC, b-BHC, d-BHC, a-Chlordane, g-Chlordane,	0.0050	0.010	0.020	0.030	0.050
p,p'-DDE, Endrin p,p'-DDD, Endosulfan sulfate, Endrin ketone	0.010	0.020	0.040	0.060	0.10

DDT/Endrin Breakdown Evaluation Standard

Pesticide Evaluation Standard	CONC (ug/mL)
Endrin,	0.020
P,P'-DDT	0.020

Appendix IX Targets

Target compound	ISMB-1 (ug/mL)	ISMB-2 (ug/mL)	ISMB-3 (ug/mL)	ISMB-4 (ug/mL)	ISMB-5 (ug/mL)
TCMX,DCB (surr)	0.0025	0.0050	0.010	0.020	0.040
Isodrin	0.0050	0.010	0.020	0.030	0.050
Chlorobenzilate	0.050	0.10	0.20	0.50	1.0
Kepone	0.025	0.050	0.10	0.20	0.50

APPENDIX B

Technical Chlordane Five-point Curve

STOCK STANDARD	TCHLOR -1*	TCHLOR -2*	TCHLOR -3*	TCHLOR -4*	TCHLOR -5*
Technical Chlordane	0.100	0.20	0.40	0.60	0.80
DCB, TCMX (surr)	0.0025	0.0050	0.010	0.020	0.040

Toxaphene Five-point Curve- 0.10, 0.25, 0.50, 1.0, 2.0ug/mL

STOCK STANDARD	TOX -1*	TOX -2*	TOX -3*	TOX -4*	TOX -5*
Toxaphene	0.10	0.25	0.50	1.0	2.0
DCB, TCMX (surr)	0.0025	0.0050	0.010	0.020	0.040

PCBs as Aroclors

AR1660 Standards

Calibration Std	AR1016(ug/mL)	AR1260(ug/mL)	surrogates
AR1660-1	0.10	0.10	0.0025
AR1660-2	0.20	0.20	0.0050
AR1660-3	0.50	0.50	0.010
AR1660-4	1.0	1.0	0.020
AR1660-5	2.0	2.0	0.040

Single Point Aroclor Calibration Standards

Calibration Standard	Single Pont Concentration (ug/mL)	Surrogate Concentrations (ug/mL)
AR1221	1.0	0.020
AR1232	1.0	0.020
AR1242	1.0	0.020
AR1248	1.0	0.020
AR1254	1.0	0.020

If required, five point curves for AR1221, AR1232, AR1242, AR1248, and AR1254 are prepared at the same concentrations as the AR1660 curve.

Appendix C

608/8081/8082 METHOD SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	None; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days (after leaching procedure)	40 days

*Storage temperature is 4C with a control criteria of less than 6C with no frozen samples

EXTRACTION

Aqueous: Approximately 1L of sample (contents of container) using continuous or separatory funnel extraction at pH 5-9 with methylene chloride; exchange to hexane and concentrate to final volume of 10mL
Soil/Solids: Approximately 30g of sample using sonication with 1:1 acetone/hexane or 1:1acetone/methylene chloride; Concentrate to final volume of 10mL in hexane
Wastes: Approximately 1g of sample diluted to final volume of 10mL with hexane

ANALYSIS

Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external or internal standard calibration

SEQUENCE-600-series

Endrin/p,p'-DDT breakdown evaluation (daily-every 24 hours)
Initial Calibration- 3 point single peaks compounds 1 point all Aroclors 1 point toxaphene 1 point technical chlordane
Initial Calibration Verification (ICV)
Sample analyses
Continuing calibration verification (CCV)-daily-every 24 hours Single peak compounds AR1660
RL standard(optional; required by state or client QAP)
Sample analyses

Sequence continues until all samples have been analyzed or the CCV fails the acceptance criteria. If a multi-peak target compound is detected, the extract is reanalyzed with a 3-point curve.

SURROGATE(S):
Tetrachloro-m-xylene- 0.20ug/L
Decachlorobiphenyl-0.20ug/L

BATCH QC
Method blank
LCS/LCSD- full target of single peak analytes @ 0.20ug/L
MS/MSD- full target of single peak analytes @ 0.20ug/L

Appendix C

SEQUENCE-8000-series

STANDARD/SAMPLES
Endrin/p,p'-DDT breakdown (every 12 hours)
Initial Calibration- 5 point single peaks 5 point Ar1660(note1) 1 point Toxaphene 1 point tech Chlordane 1 point remaining Aroclors
Up to twenty sample extracts
Endrin/p,p'-DDT breakdown (every 12 hours)
Continuing calibration check midpoint single peaks and midpoint Ar1660(note1)
RL Standard(optional)-lowest point on the calibration curve if required by state or client QAP
Up to twenty sample extracts
Continuing calibration check-midpoint single peaks and midpoint Ar1660(note1)

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All samples extract analyses must be bracketed by acceptable verification standards.

Note 1-A mixture of Ar1016 and Ar1260 will be used to calibrate and verify the response for PCBs and to verify the response for Toxaphene and technical Chlordane in the continuing calibration.

SURROGATE (S):
Tetrachloro-m-xylene- 0.20ug/L
Tecachlorobiphenyl-0.20ug/L

BATCH QC
Method blank
LCS- LQM subset
MS/MSD- LQM subset

Parameter	Aqueous(ug/L)	Soils(ug/kg)
Lindane	0.20	6.0
Aldrin	0.20	6.0
Heptachlor	0.20	6.0
Dieldrin	0.50	15
Endrin	0.50	15
p,p'-DDT	0.50	15

Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
p,p'-DDT and Endrin breakdown check -required for 8000-series, recommended for 600-series	Initially and every 12 hours	% breakdown of both compounds less than 20%	-re-analyze check solution -perform injector port and/or column maintenance and re-analyze
Initial Calibration- 600-series:3 point minimum with lowest point at RL 8000-series:5 point minimum with lowest point at RL	Initially prior to sample analysis, when major instrument maintenance performed, or when CCV fails	**600-series:** 1) RSD of each target <= 10%; OR 2) plot regression curve CC>=0.99 for each target **8000-series:** 1) RSD of each target <= 20%; OR 2) plot regression curve CC>=0.99 for each target (see previous page for exceptions)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification(CCV)	After every ten to twenty sample analyses and at the end of the sequence if external standard calibration is used	**600-series:** <=10% Difference/Drift **8000-series:** Percent difference or drift <= 15% (see previous page for exceptions) Retention time of CCV must fall within the daily retention time window	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards

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Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Method blank	Per batch	All targets reported less than RL in Section 5 of the STL-SL LQM	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in LQM
Lab control sample (LCS)- Subset of targets in STL SL LQM	Per batch (If MS/MSD cannot be performed , the LCS must be performed in duplicate)	Recoveries within STL-SL LQM Section 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in LQM
Matrix spike(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within STL-SL LQM Section 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in LQM
Internal Standard Response	All samples, method blanks, and QC	Response with a factor of two from the midpoint standard in the initial calibration sequence	-Evaluate chromatogram and integrations. -Reanalyze or dilute and reanalyze -Flag data
Surrogates	All samples, method blanks, and QC	Recoveries within STL-SL LQM Table 5 limits See section 11.1.1. for specifics	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in LQM
Reporting limit(RL) standard-standard at the reporting limit used to verify sensitivity of the instrument	Daily(optional-see specific state or client requirements for frequency)	Detected with reasonable sensitivity	-Reanalyze RL standard -Remake and reanalyze RL standard -Perform instrument or column maintenance, recalibrate, and reanalyze associated samples

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Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Retention time window determination	See guidance in STL-SL SOP AN66	See guidance in STL-SL SOP AN66	Use guidance in STL-SL SOP AN66: *Determination of Retention Time Windows in Gas Chromatographic Analyses*
Initial demonstration of Capability (the analyst has to perform the IDOC for either of the analogous 600 or 8000 series methods- not both)	Per work group or analyst	Within the 600- or 8000-series method limits (see STL-SL SOP CA92)	-Reanalyze QC sample for the targets that failed to meet the criteria (see STL-SL SOP CA92)
Method detection limit(MDL)	See STL-SL SOP CA90	Evaluate data using criteria STL-SL SOP CA90	-Evaluate data. Check calculations. -Reanalyze MDL samples.

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Approval Signature: R. Wayne Robbins	
Title : Corporate QA Manager	Date : 1/14/99

CHLORINATED HERBICIDES
(615 and 8151A)

1.0 SCOPE AND APPLICATION

1.1 This procedure can be used to determine the concentration of various chlorinated herbicides in water, groundwater, leachate, soil, sediment, and waste samples extracts. Table 1 lists the target compounds and the retention times for each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria are given in Section 5 of the current revisions of the Savannah Laboratories' quality assurance plans.

2.0 SUMMARY OF METHOD

2.1 A known volume or weight of sample is extracted in accordance with SL SOP EX45. The extracted chlorinated herbicide methyl derivatives are analyzed by gas chromatography (GC) configured with dual capillary columns and dual electron capture (EC) detectors. This configuration allows for simultaneous detection and confirmation of the herbicides. Identification of the target compounds in samples is done by comparing the retention times of the peaks with standards analyzed under the same GC conditions.

GC/MS confirmation may be employed if the target compound concentration is sufficiently high or if the extract is concentrated to an appropriate final volume. The esterified extract must be used for the GC/MS confirmation-do not use the 8270 extract.

2.2 This SOP is based on the guidance in SW-846 Method 8000B and Method 8151A and EPA Method 615.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and eye protection(lab glasses or face shields), must be worn. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

3.4 Diethyl ether is a flammable solvent and it must be used in a well-ventilated hood or extraction area. The solvent vapors will tend to accumulate along the floor. High concentrations of diethyl ether can cause drowsiness, dizziness, and headache.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. The glassware must be scrupulously cleaned (SL SOP AN60: Glassware Cleaning Procedures). All of the materials and reagents must be demonstrated to be free from contaminants by the analysis of reagent blanks (method blanks). Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences. The extraction procedure described in SL SOP EX45 has several steps that are designed to eliminate or minimize interferences due to sample matrix.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Aqueous samples are routinely collected in 1-L amber glass containers equipped with Teflon-lined caps. No preservative is required. The sample is iced at the time of collection and stored at 4C (less than 6C with no frozen samples) in the lab. Sample must be extracted within seven (7) days of collection and the extract must be analyzed within forty (40) days of extraction.

TCLP leachate samples are stored in the same manner as aqueous field samples. The extraction must be performed within seven(7) days of the leaching procedure and the extract analyzed within forty(40) days of extraction

5.2 Soils, sediments, sludges, and wastes are collected in glass containers equipped with Teflon-lined caps. The routine container is 500-mL glass. Larger or smaller containers may be supplied. No preservative is required. The samples are iced at the time of collection and stored at 4C (less than 6C with no frozen samples) in the lab. The hold time for herbicides in solid and non-aqueous matrices is 14 days from the date of collection. The extract must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph equipped with dual electron capture detectors and automatic liquid samplers.

6.2 Recommended Columns:

J&W DB-5 fused silica column, 30M x 0.53mm ID, 1.5um film
J&W DB-608 fused silica column, 30M x 0.53mm ID, 0.83um film

6.3 Data system compatible with the GC and capable of detecting and storing chromatographic data. Nelson 2600 or equivalent

6.4 Autosampler vials, septa, and caps

6.5 Microsyringes- 10-uL, 25-uL, 50-uL, 100-uL

7.0 REAGENTS

Hexane: Pesticide grade or equivalent

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all neat compounds or stock solutions. All preparation steps must be in accordance with SL SOP AN41:*Standard Material Traceability.*

Preparation of the Calibration Standards

If the stock standard is prepared using the free acid form, no correction is required for the concentration. The concentration of the standard is based on the weight of free acids added per unit volume prior to derivitization. A stock standard prepared from neat herbicide acids has to be derivitivized prior to analysis. If a herbicide standard is purchased as a neat methyl ester, the concentration of the standard must be corrected to the free acid concentration. See SL SOP AN43: Standard Preparation for guidance on the preparation of standards.

The calibration standards must be corrected to the weight of the acid. This will eliminate the need to correct the final concentration of the sample. The correction factors are given in Table 2.

The stock standards are prepared in hexane from either the stock standard mixes purchased from vendors or from the individual stock standards prepared in-house. An example of the preparation of the calibration standard is given in Table 3

9.0 SAMPLE PREPARATION

Sample preparation steps are given in SL SOP EX45.

10.0 PROCEDURE

10.1 GC Conditions

The columns and analytical conditions listed in this section are given for guidance. The lab must document the actual columns and conditions used for each analysis in the instrument maintenance, data system, or on the sample log.

10.1.1 Recommended Columns:

J&W DB-5 fused silica column, 30M x 0.53mm ID, 1.5µm film
J&W DB-608 fused silica column, 30M x 0.53mm ID, 0.83um film

The columns are connected to a single injection port with either a glass Tee-splitter or a glass y-splitter. This configuration allows for the simultaneous analysis and confirmation of the target compounds.

Carrier gas flow: He at approximately 7mL/min (per column)
Make-up gas flow: N2 at approximately 25mL/min (per detector)

10.1.2 Suggested GC temperature programs

APP 9 temperature program:
Initial temperature: 170 C
Initial hold time: 4 minutes
Program rate: 8 C / minute
Final temperature: 280 C
Final hold: 2 minutes
TOTAL TIME: 20 minutes

8151 Full List temperature program
Initial temperature: 100 C
Initial hold time: 2 minutes
Program rate: 8 C / minute
Final temperature : 280 C
Final hold: 2 minutes
TOTAL TIME: 27 minutes

10.1.3 Heated zone temperatures

Injector: 240-260 C
Detector: 300C

10.1.4 Injection volume: 2-4uL (1-2-uL per column)

10.2 Initial Calibration
The external standard calibration technique is routinely employed for the determination of the concentration of herbicides. The lab also has the option of using internal standard calibration. Pentachloronitrobenzene (PCNB) may be a suitable compound to use as an internal standard

10.2.1 Prepare and inject the calibration standards using the guidelines listed in Section 8 of this SOP. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in Section 10.3.

Note that the following offers two (2) options for calibration and quantitation -- average RF or regression curve. Only one need be chosen per analyte.

When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

10.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed?

>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?

>Are the peaks symmetrical? Is there tailing or fronting?

>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.3 Average RF/CF Option:

Calculate the response factor of each calibration standard, the average response factor and the relative standard deviation using the following equations:

Response Factor-external standard

$$X_{RF} = \frac{mg/mL}{area} = \frac{mg}{mL\ area}$$

Note that the inverse relationship can also be used to evaluate the response of the detector. Dividing the area of the standard by the concentration is referred to as the calibration factor.

Response factor-internal standard

$$RF = \frac{Ac}{Ais} \otimes \frac{Cis}{Cc}$$

where

Ac = area of the target compound
Ais = area of the internal standard
Cc = concentration or mass on-column of the target compound (ug/mL)
Cis = concentration or mass on-column of the internal standard (ug/mL)

Average Response Factor

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 + \ldots RFn}{n}$$

Relative Standard Deviation

$$\% RSD = \frac{\text{standard deviation}}{\overline{RF}} \otimes 100$$

and

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

Where

RF_i = response factor of the individual calibration level
$\overline{RF}$ = average response factor

Initial Calibration Criteria:

600-series: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear and the average response factor (or calibration factor) may be used for quantitation.

8000-series:If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear and the average response factor (or calibration factor) may be used for quantitation.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average response factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

10.2.4 Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPs for other exceptions to using non-linear curve fitting.

10.3 Calibration Verification

Calibration is verified every 24 hours for 600-series methods and every 12 hours or 20 samples, whichever is more frequent, for 8000-series methods by the analysis of continuing calibration check standards. Note that the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive for the compound(s) failing high are detected, these samples may be reported.

10.3.1 Analyze the mid-level standard(s). Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration:

$$\%Drift = \frac{result - expected}{expected} \times 100$$

Where
result = concentration or nanograms on-column of the calibration check standard quanted against the curve
expected = true concentration or nanograms on-column of the calibration check standard

$$\%Difference = \frac{RF_{init} - RF_{cont}}{RF_{init}} \times 100$$

Where
RF_{init} = average response factor from the initial calibration curve
Rf_{cont}= response factor from the continuing calibration standard

---

10.3.2 Continuing Calibration Verification Criteria

If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

600-series: If the percent drift or percent difference is less than or equal to 10%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

8000-series: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor is used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average response factor from the initial calibration can be used for quantitation if the average %drift or %difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

External Standard CCV: Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

10.4 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the SOP Summary after Section 15.

10.4.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks (hexane) may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.4.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. For the single point multicomponent products, the extract should be diluted until the area is no more than a factor of two above the area of the single point standard (see section 11.1 for quantitation of multi-peak target compounds).

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used (*F34 or *F42) or qualification in a case narrative provided to the client. For TCLP analyses, every effort should be made to achieve the regulatory level without substantial instrument overload.

For clients who demand we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve (i.e., a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client). Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SL SOP AN66: Determination of Retention Time Windows for Gas Chromatographic Analyses. If internal standard calibration is used, the determination of absolute retention time windows is not required. Relative retention times, as described in Section 11.4, are used to identify the target compounds.

11.0 DATA ANALYSIS/CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times), the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration, and the integration of the peaks. Manual integration must be documented in accordance with SL SOP AN65. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data.

The judgement and experience of the analyst and his/her colleagues is an important part of the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks correctly integrated?
- Are there co-eluting peaks or matrix interferences?

11.1 Qualitative analysis

Identification of the target compounds is based on retention time. The analyst should scan the sample chromatogram for the target compounds on the primary analytical column. The analyst should use the retention time (RT) window calculated around the CCV as guidance for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds to help gauge possible shifts in the RT of the target compounds. See SL SOP AN66.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peaks areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (10.3.2).

11.1.1 The surrogate should be evaluated first to check for shifts in retention times and to evaluate the surrogate recovery.

The extract contains the surrogate Dichloroacetic acid (DCAA). The recovery criteria are given in Section 5 of the SL QAPs.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Generally, it is expedient to calculate the recovery on one chromatogram. If this recovery is acceptable, the other channel is not evaluated for recovery. If interferences are suspected, evaluate recovery on the other channel. Note that given this nature, an extract is considered acceptable if one of the 2 potentially calculated recoveries is acceptable.

NOTE: If the recovery of the surrogate is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to section 13 of the current QA Plan regarding this issue.

11.1.2 Label and calculate the concentration of each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences and the overall shapes of the peaks.

If the concentration is below the detection limit, the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.3.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds to help gauge possible shifts in the RT of the target compounds.

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than 40%, the presence of the target compound is confirmed and the lower concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A large relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. If interference is detected, flag the result to note the disparity between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

The following table summarizes the general guidance for the evaluating of chromatographic data. The table assumes that the calibration criteria have been met and that the sample has acceptable associated surrogate and lab spike recoveries.

PEAK INFORMATION	ACTION	REPORT*
No peaks found on primary or secondary column		Report < RL
Peak found within RTW on primary column	Peak is tentatively identified as the target	If concentration < RL, report < RL If concentration > RL, evaluate confirmation column.
Peak found within RTW on confirmation column	Peak is confirmed as the target	If concentration < RL, report <RL If concentration >RL, calculate %RPD -if %RPD <40%, report lower concentration of primary and confirmation analyses. -if %RPD >40%, flag result to note the disparity Case narrative or note to PM may be required for complex matrices.

*RL may be the SL Reporting Limit in Table 5 of the CQAP or may be defined by the client QAP or contract.

The analyst must clearly show how the reported sample results were determined.

11.1.4 Identification "Tools"

Analysis by GC/MS (scan or SIM) may be used to confirm the presence of chlorinated herbicides (see SL SOP SM06: *Guidelines for SIM Analysis by GC/MS.)* The herbicide extract must be used to perform the GC/MS confirmation-unesterified extracts (e.g., the GC/MS extract) are not suitable for herbicide analysis or confirmation.

11.1.4.1 Relative Retention Time

The retention time of a surrogate compound provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions.. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification.

11.1.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the sinitial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience. The extract must be fortified with herbicides as methyl esters-unesterified herbicides are not amenable to GC analysis.

11.2 Calculations-External Standard

Aqueous/Liquid Samples

If the regression curve option is used, the sample concentration is calculated:

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{V}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

If the response factor option is chosen for quantitation:

$$Concentration(ug/L) = \frac{ug/mL}{response} \otimes response \otimes \frac{F \otimes DF}{V}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

Soils/Solids Samples

Regression Curve

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
Ccurve = concentration of analyte from curve (ug/mL)
F = final volume of the extract (mL)
DF = dilution factor
W = weight of sample extracted (kg)
solids = (percent solids)/100

If the response factor option is chosen for quantitation:

$$Concentration(ug/kg, dw) = \frac{ug/mL}{response} \otimes response \otimes \frac{F \otimes DF}{W \otimes solids}$$

where
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)
W = weight of sample extracted (kg)
solids = (percent solids)/100

11.3 Internal Standard Calibration

Aqueous Samples

$$Concentration(ug/L) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{V} \otimes DF$$

where

Ac = area of the target compounds
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
V = volume of sample extracted (l)
DF =dilution factor

Soil Samples

$$Concentration(ug/kg, dw) = \frac{Ac}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{FV}{W \otimes solids} \otimes DF$$

where

Ac = area of the target compounds
Ais = area of the internal standard
Cis= concentration of the internal standard (ug/mL)
RRFavg = average response factor of the target compound from the ICAL
FV = final volume of the extract(mL)
W =weight of sample extracted (kg)
Solids = (percent solids)/100
DF =dilution factor

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The **analytical batch** is discussed in SL SOP AN02: *Analytical Batching*, and these criteria are summarized in the SOP Summary included in this SOP. Calculation of QC is also given in AN02.

12.2 The **method detection limit (MDL)** is defined as the concentration of the an analyte that can be measured with a 99% confidence that the result is greater than zero. See SL SOP CA90: *Procedure for Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items are included in this revision. See Section 10 of the current SL QAP.

14.0 TROUBLESHOOTING

No items are included in this revision.

15.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

2. *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office: Washington, DC, July 1, 1988.

3. Savannah Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*, current revision.

METHOD SUMMARY

HOLD/STORAGE

Parameter	Aqueous	Soils/Solids/Wastes
Routine Container	1-L amber glass fitted with Teflon-lined cap	500-mL or smaller glass fitted with Teflon-lined cap
Preservative	None	None
Hold time	7 days from date of collection; 40 days from date of extraction	14 days from date of collection; 40 days from date of extraction
Storage	4C (less than 6C with no frozen samples) from collection to analysis	4C (less than 6C with no frozen samples) from collection to analysis

EXTRACTION

Aquoues-500mL of sample; separatory funnel extraction at pH >12 with diethyl ether followed extraction at pH<2 with diethyl ether; esterify and dilute to 10mL final volume with hexane

Soils- acidify 30g of sample mixed with acidified sodium sulfate; sonication or Soxhlet extraction with 1:1methylene chloride/acetone; concentrate and hydrolyze with KOH, treat same as aqueous sample from this point forward.

SEQUENCE

615

Initial Calibration Standards
Client samples analyzed until 24 hour clock expires
Calibration Verification standard – mid-level concentration
Client samples analyzed until 24 hour clock expires

8151

Initial Calibration Standards
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration

The sequence continues until all samples are analyzed or until the CCV standard fails to meet the acceptance criteria.

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration- 5 point minimum with lowest point at RL	Prior to sample analysis or when CCV fails	1) RSD of each target <= 20%(8151) or 10% RSD (615); OR 2) plot regression curve (CC>=0.99) (see Section 10.2 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification(CCV)	After every twenty sample analyses (or 12 hours) and at the end of the sequence	8151: Percent difference or drift <= 15% 615: Percent difference or drift <= 10% (see Section 10.3 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in Table 5 of the SL CQAP	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Lab control sample (LCS)- Subset of targets in SL CQAP	Per batch	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix spike(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze .
Surrogate	All samples, method blanks, and QC	Recoveries within SL CQAP Table 5 limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Reporting limit(RL) standard-lowest level calibration standard	Daily-required for Fla DEP	Detected with reasonable sensitivity	-Reanalyze RL standard -Remake and reanalyze RL standard -Perform instrument or column maintenance, recalibrate, and reanalyze associated samples
Initial Demonstration of Capability (IDOC)	Initially and when new analysts trained	Evaluate in accordance with method criteria	Repeat test for analytes that fail criteria
Method Detection Limit (MDL)	See SL SOP CA90	Evaluate in accordance with SL SOP CA90	Evaluate in accordance with SL SOP CA90
Retention time window determination	See SOP AN66	See SOP AN66	See SOP AN66

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TABLE 1-"full list"		
Compound	RT COL 1	RT COL 2
Dalapon	2.90	3.35
DCAA (surrogate)	13.18	13.41
MCPP	13.56	14.08
Dicamba	13.66	13.63
MCPA	14.30	14.44
Dichlorprop	14.94	15.36
2,4-D	16.10	15.89
2,4,5-TP (Silvex)	18.27	19.22
2,4,5-T	20.49	20.33
Dinoseb	21.00	23.06
2,4-DB	22.81	23.36

COL1 = DB608 COL2 = DB-5

TABLE 1A-"short list"		
Compound	RT COL 1	RT COL 2
DCAA (surrogate)	5.38	5.75
2,4-D	7.65	7.61
2,4,5-TP (Silvex)	8.80	9.31
2,4,5-T	9.68	9.74

COL1 = DB608 COL2 = DB-5

TABLE 2

HERBICIDE MOLECULAR WEIGHTS AND CORRECTION FACTORS

Herbicide acid	MWacid	MWester/ether	Correction factor
2,4-D	221.04	235.07	0.940
Dalapon	142.97	157.00	0.911
2,4-DB	249.09	263.12	0.947
Dicamba	221.04	235.07	0.940
Dichloroprop	235.07	249.09	0.944
Dinsoeb	240.22	254.24	0.945
MCPA	200.62	214.65	0.935
MCPP	214.65	228.67	0.939
2,4,5-TP(Silvex)	269.51	283.54	0.951
2,4,5-T	255.48	269.51	0.948
DCAA	205.04	219.07	0.936
Picloram	241.48	255.51	0.945
Pentachlorophenol	266.35	280.37	0.950

Example Calculation

$$CF(2,4-D) = \frac{Wacid}{Wester} = \frac{221.04}{235.07} = 0.94$$

If the standard is expressed as mass of ester per volume, convert the concentration to he acid form by multiplying by the correction factor (CF).

Table 3-Example Standard Preparation "Recipes"

"Short List" Herbicide Intermediate Standard

STOCK STANDARD	VENDOR/ PART NO.	CONC. (ug/mL)	mL of stock	fvol (mL)	conc (ug/mL)
Herbicides	Ultra HBM-815M	100	1.0	10	10
DCAA	Ultra PPS-161	100	1.0		10

Solvent is hexane

"Short" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
"short" list herbicide intermediate std	10	50	100	200	250	500	750	1000

* microliters of intermediate standard to 10mL of hexane. Seven standards are prepared and analyzed but only 5 are used. MCPP and MCPA are difficult to calibrate at low concentrations.

"Short" list Calibration Standards

Target Compounds	1*	2*	3*	4*	5*	6*	7*
DCAA, 2,4,-D, 2,4,5-TP (Silvex), 2,4,5-T	0.05	0.10	0.20	0.25	0.50	0.75	1.0

* ug/mL

"Full" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
Ultra HBM-8150M		5	10	12.5	20	50	67	100
Dicamba, 2,4,5-TP (Silvex), 2,4,5-T	10							
2,4-D, 2,4-DB, Dichlorprop	100							
Dinoseb	50							
MCPA, MCPP	10000							

* microliters to 10mL hexane

Table 3-Example Standard Preparation "Recipes"

"Full" list Calibration Standards

STOCK STANDARD	CONC. (ug/mL)	1*	2*	3*	4*	5*	6*	7*
Ultra HBM-8150M								
Dicamba, 2,4,5-TP (Silvex), 2,4,5-T	10	0.005	0.010	0.0125	0.020	0.050	0.067	0.10
2,4-D, 2,4-DB, Dichlorprop	100	0.050	0.10	0.125	0.20	0.50	0.67	1.0
Dinoseb	50	0.025	0.050	0.0625	0.10	0.25	0.33	0.50
MCPA, MCPP	10000	5.0	10	12.5	20	50	67	100

*ug/mL

Approval Signature: R. Wayne Robbins
R. Wayne Robbins
Title: Corporate QA Manager
Date: 5/1/98

MERCURY: VARIAN SPECTRA AA 20

1.0 SCOPE AND APPLICATION

This method describes the cold-vapor atomic absorption procedure for the determination of mercury in liquids, groundwaters, soils and sludges.

The practical quantitation limit for this method is 0.2 µg/L in fresh waters or in saline waters, and 0.010 mg/kg in solid matrices.

2.0 SUMMARY

This method is based on the absorption of characteristic radiation at 253.7 nm by mercury vapor. After digestion, the mercury is reduced and aerated from solution in a mixing coil. The mixture passes through a gas/liquid separator and then vapor passes through a flow cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. This procedure is adapted from Methods 7470 and 7471 from SW-846 (2).

3.0 INTERFERENCES

Potassium permanganate is added to eliminate the possibility of sulfide interference and digested organic.

Chlorine is known to interfere. Because chlorides are oxidized to free chlorine during the oxidation step, addition of extra permanganate may be necessary for chloride-containing samples.

During digestion, sample bottles are left open so free chlorine can escape. Also, free chlorine can be reduced by using an excess of hydroxylamine sulfate reagent (25 mL).

4.0 APPARATUS AND MATERIALS

Varian Spectra AA 20: This provides a mount for the absorption cell and mercury lamp.

Mercury hollow cathode lamp

Absorption cell: The cell is 17½ cm long with quartz end windows.

VGA 76 - Vapor generation accessory

Mixing coil

Pump tubing: Tygon tubing is used to pass the sample from the test tube through the mixing coil into the gas/liquid separator.

Volumetric glassware

Printer

5.0 REAGENTS AND STANDARDS

DI water: DI water is monitored for impurities. Conductivity is checked daily and must be < 1 μmho in order to be used.

Nitric acid, concentrated: reagent grade

Sulfuric aid, concentrated: reagent grade

Hydrochloric acid, concentrated: reagent grade

Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated HCL to one volume of concentrated HNO_3.

Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 50 g of $KMnO_4$ in 1000 mL of DI water.

Sodium chloride-hydroxylamine sulfate solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine sulfate in DI water in a 1-L volumetric flask and dilute to volume.

Stannous chloride: Add 100 g of stannous chloride in DI water in a 500-mL volumetric flask, add 125 mL hydrochloric acid, concentrated, and dilute to volume.

Potassium persulfate, 5% solution (w/v): Dissolve 50 g potassium persulfate in 1000 mL of DI water.

Commercial stock standard, (Baker) mercury, 1000 ppm

Mercury intermediate stock standard, 10 ppm: Add 1 mL stock standard, 1000 ppm, and 2.5 mL nitric acid to some DI water in a 100-mL volumetric flask. Dilute to volume with DI water.

Mercury intermediate working standard 0.05 ppm: Add 5 mL intermediate stock standard and 2.5 mL nitric acid to a 1-L volumetric flask and dilute to volume.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Collect a representative sample in a plastic red-dot (nitric acid) bottle. Check pH in laboratory and if necessary, acidify the sample to pH < 2 with nitric acid.

Collect soils in a plastic bottle and cool samples to 4° C.

Holding time is 28 days from date of sampling.

Holding time for CLP (3) samples is 26 days from date of sample receipt.

7.0 PROCEDURE

7.1 Calibration Standard Preparation

Transfer 0.2-, 0.4-, 1.0-, and 5.0-mL portions of intermediate working standard to a series of 125-mL glass bottles. Add DI water from a graduated cylinder to each bottle to make a final volume of 50 mL. This results in working standard concentrations of 0.2, 0.4, 1.0, and 5.0 μg/L mercury. Shake well and add 2.5 mL of concentrated H_2SO_4, 1.25 mL of concentrated HNO_3, and 7.5 mL of $KMnO_4$ solution and let stand at least 15 min. Add 4 mL of potassium persulfate and heat for 2 h in a water bath at 95° C. Cool and add 3 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. When the solution has been decolorized (excess permanganate reduced), place in test tubes onto autosampler.

7.2 **Sample Preparation**

Liquid samples: Add 50 mL of sample or a dilution brought to 50 mL to a 125-mL glass bottle. Add 1.25 mL HNO_3, 2.5 mL H_2SO_4, and 7.5 mL of $KMnO_4$ solution to each sample. Shake well after each addition. Be sure the purple color persists for at least 15 min. If not, add up to three times more $KMnO_4$ solution. Then add 4 mL of potassium persulfate to each sample, shake well, and place the samples in a water bath at 95° C for 2 h. Remove and allow the samples to cool. Add 3 mL of hydroxylamine sulfate solution to each bottle to neutralize excess $KMnO_4$. Follow the analysis procedure below.

Soil Samples: Weigh between 0.80 and 1.00 g wet weight of sample into a 125-mL glass bottle. Add 2.5 mL DI water and 2.5 mL aqua regia. Heat for 2 min in waterbath at 95° C, cool, then add 25 mL DI water and 7.5 mL $KMnO_4$ solution to sample. Mix and heat for 30 min at 95° C. Cool and add 3 mL sodium chloride-hydroxylamine sulfate solution to reduce excess $KMnO_4$. Add 27.5 mL DI water and shake well.

Saltwater: Use 100 mL of sample in order to get a lower detection limit of 0.1 µg/L. Concentrate the sample to 50 mL by heating on a hot place in a Teflon beaker. Add 4 mL H_2SO_4, 2.5 mL HNO_3, and 0.5 mL of $KMnO_4$ solution and mix well. If the pink color disappears after 15 min, add additional amounts of $KMnO_4$ solution until the pink color persists. Usually, about 7 mL is required. Prepare two blanks in the same manner. Do not heat in water bath. Then add 1 mL hydroxylamine sulfate solution to neutralize excess $KMnO_4$. Follow the analysis procedure below.

Fish and Crustaceans: Weigh between 0.20 and 0.30 g of the sample and place into a 125-mL glass bottle. Add 2 mL H_2SO_4 and 0.5 mL HNO_3 to each sample and digest in the waterbath for 30 min at 80° C, or until completely dissolved. After samples are cooled, add 7.5 mL of $KMnO_4$, and put samples back into water bath for an additional 90 min at 30° C. Remove and cool. Add 3 mL hydroxylamine sulfate solution to neutralize excess $KMnO_4$. Blanks and QC-check standards should be treated identically. Use 0.20 to 0.40 g of EPA "Trace Metals in Fish" standard for the QC-check standard. Fish are calculated on an "as is" basis. Follow the analysis procedure below.

Prepare one blank and two QC-check standards for each 20 samples. These QC check standards are to be used as ICV/CCV and liquid lab control standards. The blank will be used as ICB/CCB and preparation blank. The blanks are prepared using 50 mL of DI water and all the reagents. The QC-check standards are prepared by using 0.5 mL of EPA QC-check sample concentrate (or equivalent) diluted to a final volume of 50 mL. The QC-check standard will serve as an initial calibration standard and continuing calibration verification standard. Treat blanks and QC-check standards as samples.

7.3 **Analysis**

CAUTION: Mercury is toxic. Insure that the exit line from the spectrophotometer is led into a absorbing media in an exhaust hood or adjacent to a vent.

Fill test tubes with digested samples and label. Fill standard tubes, calibration blank tubes and rinse bottle with appropriate solutions and label. Fill one of the VGA 76 250-mL reservoirs with DI water and the other with the stannous chloride solution. Arrange samples on autosampler tray and assign places on computer. Set instrument parameters to manufacturing specifications. Align mercury lamp and flowcell. Turn on the BGA 76. Recall program on computer. Then run calibration standards and print out the calibration curve. If curve is satisfactory (i.e., correlation coefficient ≥ 0.995), proceed in running the tray.

Usual tray setup: ICV, ICB. Then add up to 10 samples, CCV, CCB, 10 more samples, CCV, CCB, etc. At the end of the tray, run CCV, and CCB.

ICV = initial calibration verification.
ICB = initial calibration blank.
CCV = continuing calibration verification
CCB = continuing calibration blank.

These standards are defined above, under sample preparation.

The computer will calculate and print out results in µg/L as the samples are analyzed.

All EPTOX extracts, all samples that suffer from matrix interferences, and all samples analyzed as part of a delisting petition are analyzed by the method of standard addition. The standard addition method used is a single-point addition, adding 1 mL of 0.05 ppm mercury working standard to 50 mL of sample, for an added concentration of 1 µg/L..

All dilution or concentration factors must be taken into account. All soils and sludges must be appropriately qualified (*e.g.*, 0.19 mg/kg dw).

7.4 **Calculations**

Liquids -- The instrument readout is in µg/L. The normal reporting units are mg/L, therefore, the answer must be corrected for units and any dilution or concentration procedure that was used on the sample.

Example: Consider an instrument readout of 0.452 µg/L Hg.

1. If 50 mL of sample is digested and the final volume of the digested sample is 50 mL, the result would only need to be corrected for units: 0.452 µg/L * (1 mg/1000 ug). The Mercury result would be 0.00045 mg/L.

2. If 5 mL of sample is digested and the final volume is 50 mL, we have diluted the sample by a factor of 10. In this case, correct the instrument readout by a factor of ten before correcting for units. The reported answer would be 0.0045 mg/L.

Solids -- If the sample is a solid, we must consider both the weight of the sample digested and the final volume to which the sample is diluted. The instrument generated units of µg/L must be converted to mg/kg as is or mg/kg dry weight basis.

$$\frac{mg}{kg}\,dw = \frac{(X\ \mu g/L) * (V\ mL) * (1\ L/1000\ mL) * (1\ \mu g/g)/(1\ mg/kg)}{W(S)}$$

where
X = instrument readout in µg/L
V = volume of digested sample
W = wet weight of the sample in g
S = % solids (expressed as decimal equivalent (i.e., 85% solids is 0.85)

To report data as mg/kg as is, simply omit the % solids from the calculation.

8.0 QUALITY CONTROL/QUALITY ASSURANCE

The instrument is calibrated using a five-point calibration curve. The levels are as follows: 0.2, 0.4, 1.0, 3.0, and 5.0 μg/L. These standards are digested following the sample preparation procedure.

Immediately following calibration, an initial calibration verification standard (ICV) from an independent source must be analyzed. The ICV is prepared following the sample preparation procedure. Results must be within 15% of the true value. If not, the analysis is terminated and hte problem corrected before proceeding.

Calibration verification standards (CCV) should be analyzed after every 10 samples and at the end of each run and must be within 20% of the true value. Samples must be rerun that are not bracketed by calibration verification standards meeting this criterion.

Lab control samples should be processed in duplicate for each batch or for every 20 samples, whichever is more frequent. This will allow the determination of accuracy and precision. If the lab control samples do not fall within 80-120% of the true values of the metal of interest, the batch must be redigested and reanalyzed.

A calibration blank (ICB) must be analyzed inmediately following the ICV and after each calibration verification standard (CCB). The ICB and CCB must be below the practical quantitation limit.

For soil samples, digest and analyze an NIST standard reference material (NBS 1646 estuarine sediment) in duplicate for every 20 samples. the determined value must fall within 70-130% of the true value.

A matrix spike sample and a matrix spike duplicate sample are analyzed for every 20 samples. The matrix spike is at 1 μg/L mercury.

Dilute samples if they are higher in concentration than the highest standard.

9.0 REFERENCES

1. *Methods for Chemical Analysis of Water and Wastes*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983.

2. *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

3. *US EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, Multi-media, Multi-concentration Document Number ILM01.0.*

CHANGE -IN-PROGRESS ATTACHMENT

SOP Document No: ME28:06.15.99:1

SOP Description: Mercury Analysis: Leeman PS200

Approval Signature: R. Wayne Robbins	
Title: Corporate QA Manager	Date: 7/15/99

The following revisions or additions have been made to the referenced SOP.

Changes in **BOLD.** A reference to the manual digestion procedures was omitted in the 12/97 revision.

1.1 This SOP describes the procedure to determine the concentration of mercury by cold vapor atomic absorption spectrophotometry (CVAA). This method contains the analytical procedures for determination of mercury in liquids, surface and groundwaters, soils, sediments, sludges, wastes and leachates (EP or TCLP) after digestion. **The automated digestion procedures for liquids are contained in SL SOPs ME29; the manual digestion procedures for liquids and soils are contained in SL SOP ME26.**

9.0 SAMPLE PREPARATION

For the preparation of samples see the appropriate section of SL SOP ME29 **(automated preparation of liquids) or SL SOP ME26 (manual digestion procedure for liquids and soils).**

Approval Signature: R. Wayne Robbins R. Wayne Robbins	
Title: Corporate QA Manager	Date: 6/17/99

MERCURY ANALYSIS: LEEMAN PS200

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure to determine the concentration of mercury by cold vapor atomic absorption spectrophotometry (CVAA). This method contains the analytical procedures for determination of mercury in liquids, surface and groundwaters, soils, sediments, sludges, wastes and leachates (EP or TCLP) after digestion. The digestion procedures for mercury are contained in SL SOPs ME29.

1.2 The reporting limit (RL) and the accuracy and precision criteria are listed in Section 5 of the current revisions of the Savannah Labs' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF THE METHOD

2.1 This method is based on the absorption of characteristic radiation at 253.7 nm by mercury vapor. After a digestion, the mercury is reduced by the addition of stannous chloride and aerated from solution in a mixing coil. The mixture passes through a gas/liquid separator and the vapor is then passed through a drying tube. The vapor is then passed through a flow cell positioned in the light path of an atomic absorption spectrophotometer. Mercury concentration is then measured as a function of absorbance.

2.2 This method is based on the guidance provided in SW-846 methods 7000A, 7470A, 7471A, and EPA method 245.1 (Drinking Water version).

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not fully understand or that will put you or others on potentially dangerous situations.

3.2 Acid spill kits must be available. These kits must be located in a highly accessible area of the lab. Each lab must have access to a properly working shower.

3.3 The standards and reagents used in this method should be treated as potential hazards. Lab coats, gloves, safety glasses and other protective equipment must be used when preparing samples, standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are available to the analyst/chemist. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions. Each person should read and understand these sheets for all standards and reagents used before beginning this procedure.

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate the possibility of interference from sulfide and certain organic compounds.

4.2 Chlorine is known to interfere. Addition of extra potassium permanganate may be needed during the digestion of chloride containing samples. Also, the samples are not capped tightly during digestion so that excess chlorine can escape.

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain mercury. Reagent blanks (method blanks) must be analyzed as a check on contamination due to sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

5.1 Aqueous samples and TCLP/EP-TOX Leachate

5.1.1 Liquid samples are collected in 250-mL plastic or glass containers. The samples are preserved with HNO3 to a pH <2. Samples must be digested and analyzed within 28 days of collection.

5.1.2 Samples for dissolved mercury should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered. The sample is stored at 4°C (less than 6°C, but not frozen) until filtration and preservation.

5.2 Soil/Sediment/Waste Samples

5.2.1 Soil and sediment samples are collected in 250-mL or 500-mL plastic or glass containers. The samples are iced at the time of collection and stored at 4C (less than 6C but not frozen) until the time of digestion and analysis. Samples must be digested and analyzed within 28 days of collection.

6.0 APPARATUS AND MATERIALS

6.1 Leeman PS200 or other suitable automated mercury analyzer with data system and printer

6.2 Nitrogen gas supply and appropriate fittings

6.3 Pump tubing of appropriate sizes for use on the PS200

6.4 Volumetric glassware for making standards and reagents

6.5 Test tubes of the two sizes to fit the PS200 autosampler

7.0 REAGENTS

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with SL SOP AN35.

7.2 Nitric Acid (HNO3), concentrated-reagent grade

7.3 Hydrochloric Acid (HCl), concentrated-reagent grade

7.4 Rinse Water, 5% HCl-1%HNO3 - to a clean 2-L bottle add 1-L of reagent water. Carefully add 100-mL of concentrated hydrochloric acid. Carefully add 20 mL of concentrated nitric acid. Dilute to a final volume of 2-L. Other volumes may be utilized providing the reagent proportions remain the same.

7.5 Stannous chloride (SnCl2.2H2O) - reagent grade, suitable for mercury determination

7.5.1 Stannous chloride ($SnCl_2.2H_2O$) solution - to a clean 2-L volumetric flask add 100-g of stannous chloride. Add approximately 400-mL of reagent water. Carefully add 500-mL of concentrated hydrochloric acid. Add a stirring bar and stir on a stir plate until the stannous chloride is dissolved. Remove the stirring bar and dilute to volume with reagent water.

7.6 Magnesium perchlorate ($Mg(ClO_4)_2$) - used as a drying agent in the drying tube. The magnesium perchlorate should be as coarse as possible

8.0 STANDARD PREPARATION

For the preparation of standards see the appropriate section of SL SOP ME29. Note that the standards are digested in the same manner as the field samples.

9.0 SAMPLE PREPARATION

For the preparation of samples see the appropriate section of SL SOP ME29.

10.0 ANALYSIS PROCEDURE

10.1 Initial startup of the instrument

10.1.1 Before analysis begins inspect the system (pump tubes, mixing coil, gas/liquid separator) to see if any parts need to be cleaned or replaced.

10.1.2 Replace the drying tube with a freshly packed drying tube, making sure that the magnesium perchlorate is not packed too tightly. The vapors must be able to pass freely through the drying tube.

10.1.3 Fill the rinse tank with rinse water.

10.1.4 If the lamp is not already on and warmed up perform a cold-start using the COLDSTRT macro. The lamp must warm up for a minimum of 2 hours.

10.1.5 If the lamp is already on and warmed up, perform a warm-start using the WARMSTRT macro. This will give the instrument time to "exercise" the pump tubes. Allow a minimum of 20 minutes of pump time for the pump tubes to break in each day.

10.1.6 Check the aperture. If the aperture is not close to 0 (+/- 100) adjust the appropriate set screw. If the aperture must be adjusted, allow at least 10 minutes for the cell to warm up after replacing the cover.

10.1.7 Fill the stannous chloride reagent bottle with stannous chloride solution. Switch the reagent line from rinse to the stannous chloride reagent bottle.

10.2 Autosampler setup

10.2.1 Fill the standard tubes with the appropriate standards for the protocol being followed. Refer to ME26 or ME29 for the prep of these standards.

10.2.2 Fill the labeled sample test tubes with the samples and calibration verification standards in the applicable order. An example order is as follows:

ICV - Initial Calibration Verification Standard
ICB - Initial Calibration Blank
10 SAMPLES
CCV - Continuing Calibration Verification Standard
CCB - Continuing Calibration Blank
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
:
:
CCV
CCB

It is appropriate to use the liquid laboratory control samples as the CCV. The specific ID for the LCS is used to identify the CCV (i.e., 1209T-1, 1209T-2). The CCVs will bracket the samples which they were prepped with.

The preparation blank will be analyzed first. The LCS will follow immediately after the preparation blank. The samples, matrix spikes and duplicates will then follow with a maximum of 10 analyses between CCVs/CCBs. All samples and control samples will be labeled with the corresponding batch ID.

10.2.3 Enter the sample/QC IDs into the autosampler table giving each rack a unique name.

10.2.4 Load the rack(s) onto the autosampler.

10.3 Calibration of the mercury analyzer.

10.3.1 Call up the required protocol. Open a new data folder. Enter the operator ID (e.g., J.Smith).

10.3.2 Go to CALIBRATION, RESET, and reset the calibration for a new calibration

10.3.3 Go to CALIBRATION, STANDARDS, and insure that calibration standards are entered at the proper concentrations.

10.3.4 Analyze the standards, beginning with standard 1 (Blank), proceeding from lowest to highest concentration.

10.3.5 When all calibration standards have been analyzed, go to CALIBRATION, LINE CALIBRATION. If calibration is within acceptable limits (correlation > 0.995) accept the linear calibration and print the calibration curve.

10.4 Sample analysis

10.4.1 Go to AUTOSAMPLER, SETUP. Enter the Rack ID (s) and the cup numbers to be analyzed.

10.4.2 Press the SAMPLE (F8) key to begin the analysis run.

10.5 If the concentration of a sample is above the calibration range of the Hg analyzer, the sample digestate must be diluted and reanalyzed. The amount of digestate needed to prepare the desired dilution is determined from the following equation.

$$V_{digest} = (Vf_{vol})/DF$$

where

V_{digest} = volume of sample digestate used to make the dilution (mL)
Vf_{vol} = final volume of diluted sample (mL)
DF = dilution factor

Samples should be diluted with digested blank solution.

10.5.1 The dilution factor is calculated as follows:

$$DF = Vf_{vol}/V_{digest}$$

where

V_{digest} = volume of sample digestate used to make the dilution (mL)
Vf_{vol} = final volume of diluted sample (mL)
DF = dilution factor

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Aqueous and Leachate Samples

The concentration of mercury in liquid samples is calculated as follows:
(Note that results may also be reported in mg/L. Results in mg/L are reported by dividing the result below by 1000).

$$C_{sample} = C_{curve} \times DF$$

where

C_{sample} = concentration of sample (ug/L)
C_{curve} = concentration from curve (ug/L)
DF = dilution factor

The reporting limit (RL) is calculated as follows:
(Note that the RL may also be reported in mg/L. Results in mg/L are reported by dividing the result below by 1000).

$$RL_{sample} = RL_{qap} \times DF$$

where

RL_{sample} = reporting limit of sample (ug/L)
RL_{qap} = reporting limit from Table 5 of CQAP (ug/L)
DF = dilution factor

11.2 Soil/Solid Samples

The concentration of mercury in soil and solid samples is calculated as follows:

$$C_{sample} = C_{curve} \text{ x } [(F)/(W \text{ x solids})] \text{ x DF x 1mg/1000ug}$$

where

C_{sample} = concentration of sample (mg/kg dw)
C_{curve} = concentration of digest from curve (ug/L)
F = final volume of digest (L)
W = weight of sample digested (kg)
solids = (percent solids)/100
DF = dilution factor

The reporting limit (RL) for soil/solid samples is calculated as follows:

$$RL_{sample} = [RL_{qap}/(W \text{ x solids})] \text{ x DF}$$

where

RL_{sample} = reporting limit of sample (mg/kg dw)
RL_{qap} = reporting limit from Table 5 of CQAP (mg/kg)
W = weight of sample digested (kg)
solids = (percent solids)/100
DF = dilution factor

The RL is based on a 1-gram sample digested to a final volume of 50-mL.

11.3 The theoretical concentration (CT) of a spiked sample is calculated:

$$C_T = (C_s \text{ x } V_s)/(V_{sample})$$

where

C_s = concentration of the matrix spiking solution (mg/L)
V_s = volume of the matrix spiking solution added to the sample (mL)
V_{sample} = volume of the sample spiked (mL)

The theoretical concentration (CT) of a soil matrix spike is calculated:

$$CT = (C_s \text{ x } V_s \text{ x } (1\text{-L}/1000\text{-mL}))/(W \text{ x solids})$$

where

C_s = concentration of the matrix spiking solution (mg/L)
V_s = volume of the matrix spiking solution added to the sample (mL)
W = weight of sample digested (kg)
solids = (percent solids)/100

11.3.1 The concentration of mercury in the lab control and matrix spike samples is determined as in Section 11.1.1. The concentration is compared to the theoretical spike concentration and the percent recovery is calculated.

The percent recovery is calculated:

$$\%REC = [(C_{MS} - C_{sample})/(C_T)] \times 100$$

where

C_{MS} = concentration of the spiked sample (mg/L or mg/kg dw)
C_{sample} = concentration of the unspiked sample (mg/L or mg/kg dw)
C_T = theoretical concentration of the spike (mg/L or mg/kg dw)
(Assume $C_{sample} = 0$ for the LCS/LCSD)

11.3.2 The relative percent difference is calculated:

$$\%RPD = |(REC_{MS} - REC_{MSD})/((REC_{MS} + REC_{MSD})/2)| \times 100$$

where

REC_{MS} = percent recovery of the MS (or LCS)
REC_{MSD} = percent recovery of the MSD (or LCSD)

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 SL SOP AN02: Analytical Batching, Table 13.1 of the SL QAP, and the SOP Summary provide guidance on evaluating QC and sample data.

13.0 PREVENTATIVE MAINTENANCE

13.1 Pump tubing: Inspect daily and replace as needed.

13.2 Standard Autosampler Cups: Clean daily and replace as needed.

13.3 Drying Tube: Repack daily, or more often if needed.

13.4 Mixing Coil: Inspect weekly, clean and replace as needed.

13.5 Sample Probe: Inspect monthly, clean and replace as needed.

13.6 Mercury Lamp: Clean or replace as needed.

14.0 TROUBLESHOOTING

No items for this section in current revision of SOP.

15.0 REFERENCES

(1) Savannah Laboratories' *Comprehensive Quality Assurance Plan* and Savannah Laboratories *Corporate Quality Assurance Plan*, current revisions

(2) *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986 (Update III).

(3) *Methods for Analysis of Water and Waste*; U.S. EPA Office of Research and Development: Cincinnati, OH, March 1983.

Method Summary - HG analysis

HOLD/STORAGE

Container	Minimum 250mL plastic or glass bottle with a plastic or Teflon-lined lid.
Preservation	*HNO3 to pH <2 in the field. If dissolved mercury is required, filter the samples before preservation.*
Storage	Solids should be stored at 4C (<6C , but not frozen) from collection until preparation.
Hold Time	Samples may be held for up to 28 days from the time of collection.

SAMPLE PREPARATION

Samples should be prepared in accordance with ME26 or ME29.

ANALYTICAL SEQUENCE

Instrument Startup	Turn on the mercury analyzer according to the instrument manufacturer's recommendations. Allow the mercury lamp proper warm-up time. Inspect and change pump tubes and drying tubes as needed. Check and align lamp and cell according to the instrument manufacturers recommendations.
Initial Calibration	Beginning with the blank, calibrate with the blank and 5 standards. One standard must be at or below the PQL.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the analysis run. The ICV solution must come from a source other than the calibration source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with a concentration at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run. The CCV and ICV may be the same solution. Analyze a calibration blank after every CCV.
Detection Limit Check Solution	At the beginning of the analysis run, verify the accuracy at the PQL by analyzing a standard with a concentration at the required PQL/CRDL.
Post Digestion Spikes/Serial dilution	At a minimum of once per analytical batch, verify the absence of matrix interference by analyzing a post digestion spike and a serial dilution.

QC CRITERIA

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 blank and 5 standards Correlation > 0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within +/- 10% 245.1 = within +/- 5%	Recalibrate
Continuing Calibration Verification Standards (CCV)	At the beginning and end of the analysis and every 10 samples.	SW846 = within +/- 20% 245.1 = within +/- 10%	Terminate the analysis, correct the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the PQL/CRDL.	SW846 = terminate the analysis, correct the problem and reanalyze the previous 10 samples.
PQL/CRA standard	After every calibration but not before the ICV.	Standard should be detected.	Recalibrate.
Laboratory control sample (LCS)	One per batch of twenty samples or less	In SL Quality Assurance Plan	Redigest and reanalyze batch
Preparation Blank - SW846	One per batch of twenty samples or less	|result| < PQL.	Redigest and reanalyze batch
MS/MSD - SW846	One set per batch of twenty samples or less	%Rec = 80 - 120% %RPD = < 20%	Flag and report data
MS - 245.1	MS added to a minimum of 10% of samples	%Rec = 70 - 130%	Flag and report data
Serial Dilution Analysis (1+4 dilution)	One per batch of twenty samples or less	If sample is at least 25 times the instrument detection limit the serial dilution, corrected for the dilution factor, should agree within +/- 10% of the undiluted sample.	Evaluate the post-digestion spike.
Post Digestion Spikes	One per batch of twenty samples or less	%Rec = 85 - 115%	Check for interference source and reanalyze samples or analyze samples by MSA.

DIGESTION PROCEDURES FOR GRAPHITE FURNACE ATOMIC ABSORPTION TOTAL METALS AND TOTAL RECOVERABLE METALS IN LIQUID SAMPLES

Approved by:

R. Wayne Robbins   Sept 22, 1999

Title: STL-SL QA Manager   Date

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used to digest water, drinking water, wastewater, and TCLP and EP leachate samples prior to analysis by graphite furnace atomic absorption (GFAA). Two digestion procedures are included: total metals and total recoverable metals. Total metals is a more vigorous digestion and must be used for 7000-series methods and TCLP and EP leachates. Total recoverable metals may be used for 200-series methods.

2.0 SUMMARY OF METHOD

2.1 Total Metals-A known volume, usually 50mL, of an aqueous or leachate sample is transferred to a Teflon beaker. The sample is refluxed with nitric acid at approximately 90-95C. After the sample has digested, as evidenced by a clear digestate, the sample is brought up to the original volume with reagent water.

2.2 Total Recoverable Metals-A known volume, usually 50mL, of an aqueous sample is transferred to a Teflon beaker. The sample is refluxed with dilute nitric acid at approximately 95C. After the sample has evaporated to approximately 15mL, the sample is brought up to the original volume with reagent water.

2.3 Drinking water samples with a turbidity concentration of less than 1 NTU can be analyzed with no digestion if the required quantitation limits can be achieved with no sample preconcentration. The exception to this rule is silver, which requires sample digestion prior to analysis.

2.4 Samples filtered for the determination of dissolved metals do not require digestion if the sample

1) has a low COD(<20mg/L);
2) has a turbidity <1 NTU ;
3) is colorless with no significant odor ; and
4) is of one liquid phase and free of suspended particulates or precipitates after acidification

(40 CFR Part 136 Table 1B-note 4)

2.5 This SOP is based on the guidance in SW-846 Method 3020A and EPA Method 200.9.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The samples are digested in strong acid solutions and contain an acid concentration of 10-20% by volume. The analyst must wear protective clothing. The acids used in this procedure will destroy unprotected clothing. The analyst must wear proper eye protection. Acid can be splashed into the eyes from many sources.

3.3 The acid digestion procedures must be performed under a properly functioning fume hood. The acid fumes from the digestion can cause mild to severe respiratory problems if breathed.

3.4 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.5 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.6 Each analyst should be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in this procedure. These sheets denote the type of hazard that each reagent poses and the safe handling instructions for these compounds.

3.7 Care must be taken when handling the digestion beakers. Before handling a vessel that has been in use, check the temperature to make sure that it is not hot. Make sure that the digestion vessels are placed on a stable platform during and after the digestion. Vibrations from the hood or an unstable platform can cause the beakers to move and possibly to fall and splatter an analyst with a hot acid solution. Hot acids can cause severe skin burns and destroy unprotected clothing.

4.0 INTERFERENCES

Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion. Glassware must be cleaned in accordance with STL-SL SOP AN60: *Glassware Cleaning Procedures.*

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	ROUTINE CONTAINER	PRESERVATIVE	STORAGE/HOLD TIME
Aqueous (water)	500-mL plastic	HNO_3 to pH <2	Ambient.

Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

The pH of all preserved samples must be checked and documented upon arrival in the lab. If the pH is not within the proper range, additional acid is added to the sample to bring the pH below 2.

- Place a piece of pH paper (wide range or narrow range can be used) on a watch glass or other inert surface.

- Transfer a few drops of the sample to the pH paper and note the color change. If the pH <2, record this in the log and transfer the sample to the storage area.

- If the pH is greater than 2, contact the Project Manager using an anomaly (STL-SL SOP CA85) for approval to adjust the pH. If the Project manager approves the pH adjustment move the sample under a hood. Add 1:1 nitric acid to the sample in 1mL aliquots, checking the sample pH after each addition, until the pH <2. The volume of 1:1 nitric acid added to the sample should not exceed 1% of the total volume of the sample. For a 500-mL sample, the maximum volume of 1:1 nitric is 5mL. If more acid is required, contact the supervisor for further guidance.

NOTE: Samples that are not at pH<2 upon arrival in the lab may contain cyanide or sulfide or may be highly buffered. Working under a hood minimizes the hazard the may be caused by the evolution of hydrogen cyanide or hydrogen sulfide upon acidification of the sample. Be aware that acid/base neutralization reactions may be violent and evolve a good deal of heat.

6.0 APPARATUS AND MATERIALS

6.1 Digestion vessels: Teflon or Pyrex beakers, 150-mL and 250mL, or comparable digestion vessels (250-mL beakers are listed in the preparation steps but 125-mL or 50-mL digestion block digestion vessels can be substituted)

6.2 Watch glasses, "ribbed", to fit over digestion vessels (not required for block digestion vessels)

6.3 Hot plates or digestion block-capable of maintaining a sample temperature of 90-95C. The hot plates or digestion block are calibrated each quarter. The temperature of 50mL of water contained in a digestion vessel at the center of the hot plate or placed in the digestion block is measured and the setting is marked when the temperature of the water reaches 85C (+/- 3C) (95C ± 5C for digestion block vessels). The temperature of the material in the vessel will rise to approximately 95C +/- 5C when a watch glass is placed on top. (This procedure for calibration is adapted from EPA Methods 200.7 and 200.9 which references both aqueous and soil/solid digestions)

NOTE: The use of hot plates is listed in the preparation steps but a digestion block may be used if the same general procedures are employed. The hot plate or digestion block settings must be recorded in the maintenance log or other suitable log.

6.4 Teflon vials-25mL

6.5 Volumetric flasks- 100-mL

6.6 Graduated cylinder-50mL

6.7 Pipettes

6.8 Analytical balance

6.9 Top-loading balance

7.0 REAGENTS

7.1 Reagent water-lab generated deionized water. ASTM Type I or Type II. The conductivity must be checked in accordance with STL-SL SOP AN35: *Conductivity Checks for Laboratory Deionized Water.*

7.2 Nitric acid (HNO_3)-reagent grade.

7.3 Nitric acid solution (1:1)- Measure 500mL of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500mL of concentrated nitric acid (HNO_3) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled container suitable for storing acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed. CAUTION: HEAT WILL BE EVOLVED AS THE NITRIC ACID MIXES WITH THE WATER. THIS SOLUTION WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING.

7.4 Hydrochloric acid (HCl)- reagent grade.

7.5 Hydrochloric acid solution (1:1)- Measure 500mL of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500mL of concentrated hydrochloric acid (HCl) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled container suitable for storing acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed. CAUTION: HEAT WILL BE EVOLVED AS THE HYDROCHLORIC ACID MIXES WITH THE WATER. THIS SOLUTION WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING.

8.0 STANDARDS

The preparation of the spiking solutions must be tracked in accordance with STL-SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in STL-SL SOP AN43:*Standard Preparation*. The expiration date of all spiking solutions is 180 days from date of preparation.

The lab should purchase certified solutions from STL-SL-approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See STL-SL SOP AN43 for guidance for standard preparation.

8.1 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

where
Vi = volume of stock standard (or *initial standard*) needed to prepare the spiking solution(mL)
Ci = concentration of stock solution (or *initial standard*)(ug/mL)
Cf = concentration of spiking solution to prepare (*final concentration*)(ug/mL)
Vf = volume of spiking solution to prepare (*final volume*)(mL)

The concentration can be expressed in whatever terms the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for Ci and Cf.

8.2 GFAA Spiking Solution
This spiking solution contains the elements that are routinely analyzed by GFAA.

8.2.1 Add 1.0mL of nitric acid to a 100mL volumetric flask containing about 50mL of reagent water.

8.2.2 Add Vi of the stock standards to the flask and dilute to a final volume of 100mL with reagent water. Mix thoroughly and transfer to a labeled storage container. Prepare this solution as needed or every six months.

The following table gives a recipe for the preparation of the GFAA matrix spiking solution:

GFAA Spiking Solution

Element	*Ci(mg/L)*	*Vi(mL)*	*Vf(mL)*	*Cf(mg/L)*
Silver (Ag)	1000	0.050	100	0.50
Arsenic(As)	1000	0.50		5.0
Selenium(Se)	1000	0.50		5.0
Lead(Pb)	1000	0.50		5.0
Thallium(Tl)	1000	0.50		5.0
Cadmium(Cd)	1000	0.050		0.50
Copper(Cu)	1000	0.50		5.0
Chromium(Cr)	1000	0.50		5.0
Antimony(Sb)	1000	0.50		5.0
Nickel(Ni)	1000	0.50		5.0

9.0 SAMPLE PREPARATION

The turbidity of drinking water samples can be checked using the procedures in STL-SL SOP BA80:*Turbidity*. If the turbidity of the sample is not checked, the digestion procedure must be performed. If silver must be determined in a drinking water sample, the sample must be digested.

9.1 Total Metals-Aqueous Samples and TCLP

9.1.1 Transfer a 50-mL aliquot (or an appropriate volume diluted to 50-mL with reagent water) of a well-mixed sample to a clean 125-mL Teflon beaker or other suitable digestion vessel.

NOTE: If there is not sufficient volume to use a 50-mL aliquot, the lab can use a smaller volume of sample and bring the final digested volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digested should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

9.1.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

9.1.3 Add 0.50mL of the graphite furnace spiking solution to a 50-mL aliquot of reagent water designated as the laboratory control spikes (LCS).

9.1.4 Add 0.50mL of the graphite furnace spiking solutions to two 50mL aliquots of the client sample designated as the matrix spikes samples(MS and MSD).

9.1.5 Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the volume of sample used
- the lot number of the acids used for the digestion
- the lot numbers of the graphite furnace LCS spiking solutions and the graphite furnace matrix spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE: A DIGESTION BATCH CONSISTS OF TWENTY FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. THE BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE (LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE (IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). IF THERE IS NOT SUFFICIENT SAMPLE FOR MS/MSD, THE LCS IS PREPARED IN DUPLICATE (LCSD).

9.1.6 Add 1.5mL of concentrated HNO_3 to each sample. Cover each beaker with a watch glass (a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 15-20mL. DO NOT ALLOW THE SAMPLE TO COMPLETELY EVAPORATE TO DRYNESS!

NOTE: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

9.1.7 Remove the beakers from the hot plate and cool the beakers to room temperature. Add another 1.5mL portion of concentrated HNO_3. Replace the watch glass and continue heating the sample on the hot plate. Again, at the proper temperature, the sample should gently reflux in the beaker-do not allow the sample to boil.

9.1.8 Continue heating the sample and adding additional 1.5-mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3. If a sample requires more than 12mL of acid to digest, contact the digestion lab supervisor for guidance.

9.1.9 Evaporate the digestate (covered with the watch glass) until the volume is approximately 5 to 10mL.

9.1.10 Wash down the inside of the beaker and the watch glass with reagent water. Dilute the sample digestate to 50mL with reagent water. Transfer the digest to a labeled storage container, usually a 125-mL plastic vial.

9.1.12 Record the analyst's initials, the final volume of the sample digestate, and the date and time that the digestion was completed in the digestion logbook. The sample is now ready for analysis by graphite furnace.

9.2 Total Recoverable Metals-Aqueous Samples (for 200-series only)

9.2.1 Transfer a 50-mL aliquot (or an appropriate volume diluted to 50-mL with reagent water) of a well-mixed sample to a clean 125-mL Teflon beaker or other suitable digestion vessel.

NOTE: If there is not sufficient volume to use a 50-mL aliquot, the lab can use a smaller volume of sample and bring the final digested volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digested should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

9.2.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

9.2.3 Add 0.50mL of the graphite furnace spiking solution to a 50-mL aliquot of reagent water designated as the laboratory control spikes (LCS).

9.2.4 Add 0.50mL of the graphite furnace spiking solutions to two 50-mL aliquots of the client sample designated as the matrix spikes samples (MS and MSD).

9.2.5 Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the volume of sample used
- the lot number of the acids used for the digestion
- the lot numbers of the graphite furnace LCS spiking solutions and the graphite furnace matrix spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE: A DIGESTION BATCH CONSISTS OF TWENTY FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. THE BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE (LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE (IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). IF THERE IS NOT SUFFICIENT SAMPLE FOR MS/MSD, THE LCS IS PREPARED IN DUPLICATE (LCSD).

9.2.6 The acids added to each sample will depend of the requested analysis.

9.2.6.1 EPA 200.9
Add 1.0mL of 1:1 HNO_3 and 0.50mL of 1:1 HCl to each sample. Cover each beaker with a watch glass (a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 15mL. DO NOT ALLOW THE SAMPLE TO COMPLETELY EVAPORATE TO DRYNESS!

9.2.6.2 Other 200-series methods
Add 1.0mL of 1:1 HNO_3 to each sample. Cover each beaker with a watch glass (a watch glass is not required for the digestion block). Gently heat the beaker until the sample refluxes-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Evaporate the sample until the volume is approximately 15mL. DO NOT ALLOW THE SAMPLE TO COMPLETELY EVAPORATE TO DRYNESS!

9.2.7 Wash down the inside of the beaker and the watch glass with reagent water.

9.2.7.1 EPA 200.9: Dilute the sample digestate to 25mL with reagent water. Transfer the digest to a labeled storage container, usually a 125-mL plastic vial.

9.2.7.2 Other 200-series methods: Dilute the sample digestate to 50mL with reagent water. Transfer the digest to a labeled storage container, usually a 125-mL plastic vial.

9.2.8 Record the analyst's initials, the final volume of the sample digestate, and the date and time that the digestion was completed in the digestion logbook. The sample is now ready for analysis by graphite furnace.

10.0 PROCEDURES

The digestion procedure is described in Section 9.0. The analytical procedure is given in STL-SL SOP ME 75: *Graphite Furnace AA*.

11.0 CALCULATIONS

Calculations for the determination of metals by GFAA are given in STL-SL SOP ME75: *Graphite Furnace AA*.

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

STL-SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with STL-SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 Method 3020. *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC. (Update III).

15.3 EPA Method 200.9. *Methods for the Determination of Metals in Environmental Samples*, May 1994, Supplement 1. (EPA 600/R-94/111).

Approval Signature:	R. Wayne Robbins R. Wayne Robbins
Title: Corporate QA Manager	Date: July 6, 1998

DIGESTION PROCEDURES FOR ICP
TOTAL METALS IN SOILS, SEDIMENTS,WASTES, AND OILS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used to digest soil, sediment, waste, and oil samples prior to the analysis by ICP (SL SOP ME70).

2.0 SUMMARY OF METHOD

2.1 A known weight (1-2g) of the well mixed sample is transferred to a Teflon beaker or suitable digestion vessel. The sample is digested with aliquots of nitric acid and hydrogen peroxide to break down the organics present in the sample. After sample has digested, as evidenced by a clear, pale yellow digestate, HCl is added to give an approximate acid concentration of 1% HCl and 5% HNO3 and the sample digest is diluted to 100mL with reagent water.

A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits.

2.2 The SOP is based on the guidance in SW-846 Method 3050B.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The samples are digested in strong acid solutions and contain an acid concentration of 10-20% by volume. The analyst must wear protective clothing such as a lab coat or apron. The acids used in this procedure will destroy unprotected clothing. The analyst must wear proper eye protection such as lab glasses or face shield. Acid can be splashed into the eyes from many sources. Gloves must be worn to protect hands from acid burns.

3.3 The acid digestion procedures must be performed under a properly functioning fume hood. The acid fumes from the digestion can cause mild to severe respiratory problems if breathed.

3.4 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.5 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.6 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses and the safe handling instructions for these compounds.

3.7 Care must be taken when handling the digestion beakers. Before handling a vessel that has been in use, check the temperature to make sure that it is not hot. Make sure that the digestion vessels are placed on a stable platform during and after the digestion. Vibrations from the hood or an unstable platform can cause the beakers to move and possibly to fall and splatter an analyst with a hot acid solution. Hot acids can cause severe skin burns and destroy unprotected clothing.

4.0 INTERFERENCES

Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	ROUTINE CONTAINER	PRESERVATIVE	STORAGE/ HOLD TIME
Soils/ sediments	500-mL plastic	None	<6C but not frozen
Waste/Oils	500-mL plastic or glass*	None	ambient or <6C but not frozen

*some organic wastes may destroy plastic containers

6.0 APPARATUS AND MATERIALS

6.1 Teflon or Pyrex beakers, 150-mL and 250mL

6.2 Watch glasses, to fit over beakers

6.3 Teflon vials-25mL

6.4 Hot plates-capable of maintaining a sample temperature of 95C+/-5C. The hot plates are calibrated each quarter. The temperature of 50mL water contained in a digestion vessel at the center of the hot plate is measured and the hot plate setting is marked when the temperature of the water reaches 85C. The temperature of the material in the vessel will rise to approximately 90-95C when a watch glass is placed on top. (This procedure for hot plate calibration is adopted from EPA Methods 200.7 and 200.9, which reference both aqueous and soil/solid digestions)

6.5 Volumetric flasks- 100-mL,

6.6 Graduated cylinder-100mL

6.7 Pipettes

6.8 Analytical balance

6.9 Top-loading balance

7.0 REAGENTS

Reagents must be tracked in accordance with SL SOP AN44: *Reagent Traceability*.

7.1 Reagent water-lab generated deionized water. ASTM Type I or Type II. The conductivity must be checked daily in accordance with SL SOP AN35: *Conductivity Checks for Laboratory Deionized Water*.

7.2 Nitric acid (HNO_3)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals. Each lot of acid must be assayed in the laboratory to ensure that each particular lot can be used for trace analysis.

7.3 Nitric acid solution (1:1)- Measure 500ml of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500ml of concentrated nitric acid (HNO_3) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled container suitable for storing acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed.
CAUTION: HEAT WILL BE EVOLVED AS THE NITRIC ACID MIXES WITH THE WATER. THIS SOLUTION IS WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING.

7.4 Hydrochloric acid(HCl)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals. Each lot of acid must be assayed in the laboratory to ensure that each particular lot can be used for trace analysis.

7.5 Hydrochloric acid solution (1:1)- Measure 500ml of reagent water into a 2-L beaker. Place the beaker on a magnetic stir plate and add a Teflon stir bar to the beaker. Carefully and slowly add 500ml of concentrated hydrochloric acid (HCl) to the reagent water in the beaker on the magnetic stir plate. Transfer the reagent to a labeled storage container suitable for acidic solutions. Do not store reagents in volumetric glassware. Prepare this reagent as needed.
CAUTION: HEAT WILL BE EVOLVED AS THE HYDROCHLORIC ACID MIXES WITH THE WATER. HYDROCHLORIC ACID HAS A SUFFOCATING ODOR AND MUST BE USED UNDER THE HOOD. THIS SOLUTION IS WILL CAUSE SKIN BURNS AND DESTROY UNPROTECTED CLOTHING. PREPARE THIS SOLUTION UNDER A HOOD.

7.6 Hydrogen peroxide, 30%-reagent grade. Check for impurities by the analysis of a method blank.

8.0 STANDARDS

The preparation of the spiking solutions must be tracked in accordance with SL SOP AN41:*Standard Material Traceability*. General guidance on the preparation of standards is given in SL SOP AN43:*Standard Preparation.*

The lab should purchase certified solutions from SL-approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SL SOP AN43 for guidance for standard preparation.

8.1 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Ci \otimes Vi = Cf \otimes Vf$$

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

where

Vi = volume of stock standard needed to prepare the spiking solution (mL)

Ci = concentration of stock solution (ug/mL)

Cf = concentration of spiking solution to prepare (ug/mL)

Vf = volume of spiking solution to prepare (mL)

The concentration can be expressed in whatever terms the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for *Ci* and *Cf*.

8.2 Preparation of the ICP Matrix Spiking Solutions

8.2.1 ICP Matrix Spiking Solution 1 is a solution purchased from SPEX. The catalogue number is SPIKE-1. Store this solution at room temperature. Prepare this solution every six months or sooner if needed or required.

8.2.2 Preparation of the ICP Matrix Spiking Solution 2

Add 20-mL to 30-mL of reagent water to a clean100-mL volumetric flask. Add 1-mL of concentrated nitric acid (HNO_3) and 5-mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume.

Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of std. (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100

Dilute to a final volume of 100-mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

9.0 SAMPLE PREPARATION

This digestion procedure is used for the preparation of soil, sediment, waste, and oil samples for total metal determination by ICP (or flame AA). This digestion procedure is not suitable for some analytes that will be analyzed by graphite furnace atomic absorption (GFAA) because HCl can cause interferences during furnace atomization.

9.1 Weigh 1g to 2-g (wet weight) of a homogeneous sample into a 125-mL Teflon beaker or other suitable digestion vessel. The lab may weigh a larger aliquot equal to 1.0g of sample on a dry weight basis.

NOTE: A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits; if 0.1g is digested, the final volume of the digestate must be 10mL. If the sample weight: final volume ratio is greater than 1:100, the reporting limits will be higher than those listed in the CQAP.

9.1.1 To homogenize a soil sample, the sample may be vigorously stirred in the sample container or transferred a plastic "baggie" and thoroughly mixed by kneading the container. After the sample is homogenized, return only enough sample to the original container to fill it three fourths full. This will allow the sample to be stirred and homogenized if additional aliquots of the sample are required. Place the discarded sample in a containerized waste receptacle for disposal.

9.1.2 If the sample is a solid material, break up the solid in the baggie by hitting the sample with a hammer or other suitable crushing device. Contact the immediate supervisor if the matrix is difficult to break up or is difficult to mix.

9.2 Add 1.0-mL of the appropriate spiking solution to the designated laboratory control spikes. The LCS/LCSD are prepared by weighing 1g aliquots of the "blank soil" into labeled beakers or digestion vessels.

9.3 Add 1.0mL of the appropriate spiking solution to the designated matrix spike samples. The MS/MSD are prepared by weighing 1g to 2g of the sample chosen for the matrix spike into labeled beakers or digestion vessels. The lab may weigh a larger aliquot equal to 1.0g of sample on a dry weight basis for the MS/MSD or may use a smaller weight if the final volume of the digestate is adjusted or if higher reporting limits are acceptable.

9.4 Record the following information on the digestion log:

- date
- analyst's initials
- beaker ID#
- sample # and description
- the weight of sample used
- the lot number of the acids used for the digestion
- the lot number of the ICP spiking solutions
- the time that the digestion was started
- the SOP/method number

NOTE: THE DIGESTION BATCH CONSISTS OF TWENTY OR FEWER FIELD SAMPLES AND THE ASSOCIATED QC ITEMS. A DIGESTION BATCH IS NOT TO EXCEED 20 FIELD SAMPLES. EVERY DIGESTION BATCH WILL HAVE A METHOD BLANK, A LABORATORY CONTROL SAMPLE(LCS), A MATRIX SPIKE AND A MATRIX SPIKE DUPLICATE(IF THERE IS SUFFICIENT SAMPLE FOR THE MS/MSD). PERFORM THE LCS IN DUPLICATE IF THE MS/MSD CANNOT BE PERFORMED. THE METHOD BLANK IS PERFORMED WITH THE REAGENTS USED FOR THE DIGESTION. LAB SPIKES FOR SOIL MATRICES WILL BE PERFORMED USING 1-G ALIQUOTS OF BLANK SAND.

9.5 Add 5mL of reagent water and 5mL of concentrated HNO_3 to each beaker, mix, and cover the beaker with a watchglass.

9.6 Carefully heat the beaker until a gentle reflux is achieved-the sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the beaker. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the watch glass and the sides of the beaker and fall back into the beaker. Do not allow the samples to boil. Reflux for 10-15 minutes.

9.7 Remove the beakers from the hot plate and allow the beakers to cool to room temperature. Add 5mL of concentrated HNO_3 to each sample. Replace the watchglass and return the beakers to the hot plate. Carefully heat the beaker until a gentle reflux is achieved. Reflux the samples for 30 minutes. Do not allow the samples to boil.

9.8 Repeat the procedure in Step 9.7 with a second 5-mL portion of concentrated HNO_3 if brown fumes are given off. Repeat 9.7 until no brown fumes are given off.

9.9 Evaporate the sample digestate to approximately 10mL. Do not allow the bottom of the beaker to go dry during the evaporation. Allow the sample to cool to room temperature before continuing onto the next step.

NOTE: If the sample is still warm when the 30% H_2O_2 (hydrogen peroxide) is added in the next step, the sample may "boil over" and the entire process must be started over.

9.10 Add 2mL of reagent water to each beaker. Slowly and carefully add 3mL of 30% H_2O_2 to each beaker. It is very important to add the hydrogen peroxide slowly to prevent loss of sample due to vigorous effervescence. Return the beakers to the hot plate and heat until the effervescence subsides. Cool the beaker after the effervescence subsides.

9.11 Continue to add 30% H_2O_2 in 1-3mL aliquots to the sample digestate until the effervescence is minimal or until the general appearance of the digestate is unchanged. Warm the sample digestate after each addition of H_2O_2 on the hot plate.

NOTE: Do not add more than 10mL of hydrogen peroxide to each sample.

9.12 After the last addition of peroxide, reduce the volume of the digest to 5-10mL without boiling and without allowing the bottom of the beaker to go dry. Add 10mL of concentrated HCl to each sample digestate. Replace the watch glass and return the beakers to the hot plate and reflux the sample digestates for 10-15 minutes.

9.13 Wash down the inside of the beaker and the watchglass with reagent water. Dilute the sample digestate to 100mL with reagent water in a clean 100-mL graduated cylinder. Transfer the digestate to a labeled storage container.

9.14 Record the analyst's initials, the final volume of the sample digestate, and the date that the digestion was completed in the digestion logbook. The sample is now ready for analysis.

10.0 PROCEDURES

The digestion procedure is described in Section 9.0. The analytical procedure is given in SL SOP ME 70: *Elements by ICP*.

11.0 CALCULATIONS

Calculations for the determination of metals by GFAA are given in SL SOP ME70:*Elements by ICP*

12.0 QUALITY CONTROL/QUALITY ASSURANCE

12.1 The analytical batch consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

SL SOP AN02: *Analytical Batching* contains guidance for evaluating the QC in an analytical batch.

12.2 The lab must perform a method detection limit (MDL) study annually in each matrix in accordance with SL SOP CA90: *Procedure for the Determination of Method Detection Limit (MDL).*

13.0 PREVENTIVE MAINTENANCE

No items in this revision.

14.0 TROUBLESHOOTING

No items in this revision.

15.0 REFERENCES

15.1 *Savannah Laboratories Comprehensive Quality Assurance Plan* and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Approval Signature:	R. Wayne Robbins (signature)
	R. Wayne Robbins
Title: Corporate QA Manager	Date: June 19, 1998

ELEMENTS BY ICP
(200.7 and 6010B)

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures to determine the concentration of various elements by inductively coupled plasma (ICP) atomic emission spectroscopy. This method contains the analytical procedures for the determination of metals in surface and ground water, wastewater, soil, sediment, leachate (EP or TCLP), and waste samples after digestion.

1.2 Table 1 lists the elements that may be determined by ICP and the characteristic wavelength used for each element. The reporting limit (RL) for each element, the method detection limit (MDL) for each element, and the accuracy and precision criteria for each element are listed in Section 5 of the Savannah Labs' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan*.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis by ICP, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and exited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

2.2 This method is based on EPA Method 200.7 and SW-846 Method 6010B. Note that EPA has promulgated two versions of method 200.7-one for NPDES samples and one for drinking water. The calibration sequence for drinking water by 200.7 requires a multi-point curve with a minimum of three standards and a calibration blank.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.3 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions.

4.0 INTERFERENCES

4.1 Spectral interferences are caused by (1) the overlap of a spectral line from another element, (2) unresolved overlap of molecular band spectra, (3) background contribution from continuous phenomena, and (4) stray light from the line emissions of highly concentrated elements.

4.1.1 Spectral overlap may be compensated for by the use of inter-element correction factors.

4.1.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample digestate, by using a peristaltic pump, or by using the method of standards additions(MSA), or use of an internal standard

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Aqueous Samples

5.1.1 Liquid samples are collected in 250mL or 500mL plastic containers. The sample is preserved with HNO_3 to a pH <2. The sample must be digested and analyzed within 6 months of collection.

5.1.2 Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

5.2 Soil/Sediment Samples

Soil and sediment samples are collected in 500mL plastic containers. The sample is iced at the time of collection and is stored in the lab at 4C (less than 6C but not frozen) until time of digestion and analysis. The sample must be digested and analyzed within 6 months of collection.

5.3 TCLP or EP Toxicity Leachate Samples

The leachate is transferred to a plastic container after the extraction procedure. The sample is preserved with HNO_3 to a pH <2. The leachate sample must be digested and analyzed within 6 months of collection. If the leachate is to be analyzed for mercury, the leachate is stored at 4C (less than 6C but not frozen) until the mercury analysis is completed. Hold time for mercury is 28 days.

5.4 Waste Samples

Waste samples are collected in 500mL plastic containers. The sample must be digested and analyzed within 6 months of collection.

6.0 APPARATUS AND MATERIALS

6.1 Thermo Jarrell Ash ICAP-61, TJA- Enviro 36, TJA ICAP61E-trace, or other suitable inductively coupled plasma emission spectrometer with data system

6.2 Argon gas supply and appropriate fittings

6.3 Cooling water supply

6.4 Peristaltic pump

6.5 Volumetric flasks

6.6 Pipettes

7.0 REAGENTS

Reagents are tracked in accordance with SL SOP AN44:*Reagent Traceability.*

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with SL SOP AN35.

7.2 Nitric acid (HNO_3)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

7.4 Hydrochloric acid (HCl)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Table 2 lists the various stock standards that are used to prepare the calibration and QC standards. Certificates of analysis or purity must be received with all neat compounds or stock solutions. All preparation steps must be in accordance with SL SOP AN41: *Standard Materials Traceability.*

NOTE: Many standards are to be prepared every six months "or sooner if needed or required," "if needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

8.1 Initial Calibration Standards

8.1.1 Preparation of the Calibration Blank (ICB, CCB)

The calibration blank is reagent water that has been acidified with a mixture of HCl and HNO_3. The calibration blank is used as the initial calibration blank (ICB) and the continuing calibration blank (CCB).
The calibration blank must be prepared every six months or sooner if target elements are detected above the reporting limit.

EXAMPLE: Preparation of One Liter of the Calibration Blank

Add 500mL to 600mL of reagent water to a clean 1-L volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The Calibration Blank will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Dilute to a final volume of 1.0-L with reagent water. Store the standard at room temperature.

Larger volumes may be prepared at the discretion of the lab. The nitric acid concentration must be 1% by volume and the hydrochloric acid concentration must be 5% by volume.

8.1.2 Preparation of Calibration STANDARD 2

Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (ml)	Conc. of Cal Std (mg/L)
Silver(Ag)	1000	0.50	500	1.0
Arsenic(As)	1000	0.50		1.0
Molybdenum(Mo)	1000	0.50		1.0
Lead(Pb)	1000	0.50		1.0
Selenium(Se)	1000	5.0		10
Antimony(Sb)	1000	0.50		1.0
Thallium(Tl)	1000	5.0		10

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this standard every six months if needed or required.

8.1.3 Preparation of Calibration STANDARD 3

Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Beryllium(Be)	1000	0.50	500	1.0
Barium(Ba)	1000	5.0		10
Cadmium(Cd)	1000	0.50		1.0
Cobalt(Co)	1000	0.50		1.0
Chromium(Cr)	1000	5.0		10
Copper(Cu)	1000	5.0		10
Manganese(Mn)	1000	5.0		10
Nickel(Ni)	1000	2.5		5.0
Zinc(Zn)	1000	2.5		5.0

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this standard every six months or sooner if needed or required.

8.1.4 Preparation of Calibration STANDARD 4

Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum(Al)	10000	0.50	500	10
Iron(Fe)	10000	0.50		10
Boron(B)	1000	5.0		10
Strontium(Sr)	1000	5.0		10

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this standard every six months or sooner if needed or required.

8.1.5 Preparation of Calibration STANDARD 5

Add 200mL to 300mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Calcium(Ca)	10000	0.50	500	10
Potassium(K)	10000	1.0		20
Magnesium(Mg)	10000	0.50		10
Sodium(Na)	10000	0.50		10
Tin(Sn)	1000	5.0		10
Vanadium(V)	1000	5.0		10

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this standard every six months if needed or required.

8.1.6 Preparation of Calibration Standard SiO_2

Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volume of the stock standard given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume of Cal Std (ml)	Conc. Of Stock Std (mg/l)
Silica (SiO2)	1000	1.0	100	10

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this standard ever six months if needed or required.

8.1.7 Preparation of the calibration standards for multi-point instrument calibration.

For all drinking water samples the ICP must be calibrated with a minimum of three standards and a blank. The following standards may be used for this purpose.

With the Thermo Jarrell Ash software the Calibration Analysis and Curve-fit programs must be used to be successful with the multi-point calibration of the ICP instruments.

8.1.7.1 Preparation of the High Standard.

Add 200mL to 300mL of reagent water to a clean 1000-L volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum (Al)	10000	1.0	1000	10
Antimony (Sb)	1000	1.0		1.0
Arsenic (As)	1000	1.0		1.0
Boron (B)	1000	10		10
Barium (Ba)	1000	10		10
Beryllium (Be)	1000	1.0		1.0
Cadmium (Cd)	1000	1.0		1.0
Calcium (Ca)	10000	1.0		10
Cobalt (Co)	1000	1.0		1.0
Chromium (Cr)	1000	10		10
Copper (Cu)	1000	10		10
Iron (Fe)	10000	1.0		10
Lead (Pb)	1000	1.0		10
Magnesium (Mg)	10000	1.0		10
Manganese (Mn)	1000	10		10
Molybdenum (Mo)	1000	1.0		1.0
Nickel (Ni)	1000	5.0		5.0
Potassium (K)	10000	1.0		10
Selenium (Se)	1000	10		10
Silver (Ag)	1000	1.0		1.0
Sodium (Na)	10000	1.0		10
Strontium (Sr)	1000	10		10
Thallium (Tl)	1000	10		10
Tin (Sn)	1000	10		10
Vanadium (V)	1000	10		10
Zinc (Zn)	1000	5.0		5.0

Dilute to a final volume of 1000mL with reagent water. Store this standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.1.7.2 Preparation of the Mid-Level Standard.

Prepare as the CCV is prepared (8.3).

8.1.7.3 Preparation of the Low-Level Standard.

Prepare as the RL/PQL Check Standard (8.4).

8.2 Preparation of the Initial Calibration Verification (ICV) Solution

The ICV is analyzed after the ICP is standardized and the standardization has been checked by the analysis of the calibration standards as "unknowns"(the analysis of the calibration standards as "unknowns" must agree within +/- 5% of the true concentration). The ICV solution verifies that the instrument is measuring the target elements within specified criteria. The ICV must be prepared from stock standards that are obtained from a different source than the stock standards used to prepare the calibration standards. The second source may be from a separate vendor or from a separate lot from the same vendor.

For silica this standard may be used for the ICV and CCV standard.

8.2.1 Add approximately 400mL of reagent water to a 500mL volumetric flask. Carefully add 25mL of concentrated HCl and 5mL of concentrated HNO_3 to the volumetric flask. The solution will have an acid concentration of 5% HCl and 1% HNO_3.

8.2.2 Using a calibrated pipette, transfer 5.0mL of SPEX QC-7 and 5.0mL of SPEX QC-19 to the 500mL volumetric flask. Add 0.50mL of the 1000mg/L Tin (Sn) stock standard to the 500mL volumetric flask. The tin (Sn) stock standard must be from a separate vendor than the vendor of the stock standard used to prepare the calibration standard.

8.2.3 Dilute to volume with reagent water. Store the standard at room temperature. Prepare this standard every six months or sooner if needed or required.

8.3 Preparation of the Continuing Calibration Verification (CCV) Solutions

The CCV is analyzed after every 10 samples (with a suggested maximum of 2 hours elapsed time), to verify that the ICP is detecting the target elements within the specified criteria.

Add 200mL to 300mL of reagent water to a clean 1000mL volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
Aluminum (Al)	10000	0.50	1000	5.0
Antimony (Sb)	1000	0.50		0.50
Arsenic (As)	1000	0.50		050
Boron (B)	1000	5.0		5.0
Barium (Ba)	1000	5.0		5.0
Beryllium (Be)	1000	0.50		0.50
Cadmium (Cd)	1000	0.50		0.50
Calcium (Ca)	10000	0.50		5.0
Cobalt (Co)	1000	0.50		0.50
Chromium (Cr)	1000	5.0		5.0
Copper (Cu)	1000	5.0		5.0
Iron (Fe)	10000	0.50		5.0
Lead (Pb)	1000	0.50		0.50
Magnesium (Mg)	10000	0.50		5.0
Manganese (Mn)	1000	5.0		5.0
Molybdenum (Mo)	1000	0.50		0.50
Nickel (Ni)	1000	2.5		2.5
Potassium (K)	10000	0.50		5.0
Selenium (Se)	1000	5.0		5.0
Silver (Ag)	1000	0.50		0.50
Sodium (Na)	10000	0.50		5.0
Strontium (Sr)	1000	5.0		5.0
Thallium (Tl)	1000	5.0		5.0
Tin (Sn)	1000	5.0		5.0
Vanadium (V)	1000	5.0		5.0
Zinc (Zn)	1000	2.5		2.5

Dilute to a final volume of 1000mL with reagent water. Store this standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.4 Preparation of the RL/PQL Check Standard for ICP

The RL/PQL Check standard is analyzed at the beginning and end of each analysis sequence as a check on the sensitivity of the ICP. The elements in the RL/PQL Check standard must be detected. Note that there are separate standards for ICP and ICP-trace.

8.4.1 Preparation of RL/PQL Stock A-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Silver (Ag)	1000	0.10	100	1.0
Arsenic (As)	1000	1.0		10
Cadmium (Cd)	1000	0.050		0.50
Copper (Cu).	1000	0.25		2.5
Nickel (Ni)	1000	0.40		4.0
Lead (Pb)	1000	0.50		5.0
Selenium (Se)	1000	5.0		50
Thallium (Tl)	1000	5.0		50

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature.

8.4.2 Preparation of RL/PQL Stock B-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Aluminum (Al)	10000	0.20	100	20
Boron (B)	1000	0.50		5.0
Barium (Ba)	1000	0.10		1.0
Beryllium (Be)	1000	0.050		0.50
Calcium (Ca)	10000	0.50		50
Cobalt (Co)	1000	0.10		1.0
Chromium (Cr)	1000	0.10		1.0
Iron (Fe)	10000	0.050		5.0
Magnesium (Mg)	10000	0.50		50
Manganese (Mn)	1000	0.10		1.0
Molybdenum (Mo)	1000	0.10		1.0
Sodium (Na)	10000	0.50		50
Antimony (Sb)	1000	0.50		5.0
Strontium (Sr)	1000	0.10		1.0
Tin (Sn)	1000	0.50		5.0
Vanadium (V)	1000	0.10		1.0
Zinc (Zn)	1000	0.20		2.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature.

8.4.3 Preparation of RL/PQL Stock C-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volume of the stock standard given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume of Cal Std	Conc. Of Stock Std
Potassium(K)	10000	1.0	100	100

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.4.4 Preparation of the RL/PQL Check Solution-ICP

Add 200mL to 300mL of reagent water to a clean 500mL volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

RL/PQL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-ICP	5.0	500
Stock B-ICP	5.0	
Stock C-ICP	5.0	

Dilute to a final volume of 500*mL* with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.5 Preparation of the RL/PQL Check Standard for ICP-Trace

In addition to the solution prepared in 8.4.4, the following solution is prepared and analyzed to verify performance on the TJA-Trace.

The RL/PQL Check standard is analyzed at the beginning and end of each analysis sequence as a check on the sensitivity of the ICP. The elements in the RL/PQL Check standard must be detected. Note that there is an additional standard for the ICP-Trace.

8.5.1 Preparation of RL/PQL Stock A-Trace

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Arsenic (As)	1000	1.0	100	10
Antimony(Sb)	1000	2.0		20
Lead (Pb)	1000	0.50		5.0
Selenium (Se)	1000	1.0		10
Thallium (Tl)	1000	1.0		10

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature.

8.5.2 Preparation of the RL/PQL Check Solution-Trace

Add 200mL to 300mL of reagent water to a clean 500mL volumetric flask. Add 5.0mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

RL/PQL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-Trace	0.50	500

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.6 Preparation of the ICP Interference Check Solutions

These solutions are analyzed at the beginning and end of each analytical, as a check on the inter-element correction factors.

8.6.1 Preparation of ICP Interference Check Solution A

Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. Of Stock(mg/L)	mL of Stock Std	Final Volume(mL)	Conc. (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.6.2 Preparation of ICP Interference Check Solution AB *-ICP*

Add 100mL to 200mL of reagent water to a clean, amber 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock(mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200
Silver (Ag)	1000	0.50		1.0
Cadmium (Cd)	1000	0.50		1.0
Nickel (Ni)	1000	0.50		1.0
Lead (Pb)	1000	0.50		1.0
Zinc (Zn)	1000	0.50		1.0
Barium (Ba)	1000	0.25		0.50
Beryllium (Be)	1000	0.25		0.50
Cobalt (Co)	1000	0.25		0.50
Chromium (Cr)	1000	0.25		0.50
Copper (Cu)	1000	0.25		0.50
Manganese (Mn)	1000	0.25		0.50
Vanadium (V)	1000	0.25		0.50
Arsenic (As)	1000	0.50		1.0
Selenium (Se)	1000	0.50		1.0
Molybdenum (Mo)	1000	0.50		1.0
Thallium (Tl)	1000	0.50		1.0
Antimony (Sb)	1000	0.50		1.0
Tin (Sn)	1000	0.50		1.0

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.6.3 Preparation of ICP Interference Check Solution AB -Trace

Add 100mL to 200mL of reagent water to a clean, amber 500mL volumetric flask. Add 5mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock(mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200
Silver (Ag)	1000	0.50		1.0
Arsenic (As)	1000	0.050		0.10
Barium (Ba)	1000	0.25		0.50
Beryllium (Be)	1000	0.25		0.50
Cadmium (Cd)	1000	0.50		1.0
Cobalt (Co)	1000	0.25		0.50
Chromium (Cr)	1000	0.25		0.50
Copper (Cu)	1000	0.25		0.50
Manganese (Mn)	1000	0.25		0.50
Nickel (Ni)	1000	0.50		1.0
Lead (Pb)	1000	0.025		0.050
Antimony (Sb)	1000	0.30		0.60
Selenium (Se)	1000	0.025		0.050
Thallium (Tl)	1000	0.050		0.10
Vanadium (V)	1000	0.25		0.50
Zinc (Zn)	1000	0.50		1.0
Molybdenum (Mo)	1000	0.50		1.0
Tin (Sn)	1000	0.50		1.0

Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.7 Preparation of the ICP Matrix Spiking Solutions

8.7.1 ICP Matrix Spiking Solution 1 is a solution purchased from SPEX. Table 5 lists the components of SPEX SPIKE 1. Store this solution at room temperature. Prepare this solution every six months or sooner if needed or required.

8.7.2 Preparation of the ICP Matrix Spiking Solution 2

Add 20mL to 30mL of reagent water to a clean100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.8 Preparation of the IDL/MDL-ICP Solution

The IDL/MDL solution is used in this procedure for two purposes:

1) To determine the Instrument Detection Limit (IDL) of each target compound on a quarterly basis; and
2) To determine the Method Detection Limit (MDL) of each target compound as defined in SL SOP CA90.

8.8.1 Preparation of IDL/MDL Stock A-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Silver (Ag)	1000	0.10	100	1.0
Arsenic (As)	1000	1.0		10
Cadmium (Cd)	1000	0.10		1.0
Copper (Cd)	1000	0.10		1.0
Nickel (Ni)	1000	0.20		2.0
Lead (Pb)	1000	0.40		4.0
Selenium (Se)	1000	1.0		10
Thallium (Tl)	1000	1.0		10

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.8.2 Preparation of IDL/MDL Stock B-ICP

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Aluminum (Al)	10000	0.10	100	10
Boron (B)	1000	0.25		2.5
Barium (Ba)	1000	0.040		0.40
Beryllium (Be)	1000	0.010		0.10
Calcium (Ca)	10000	0.050		5.0
Cobalt (Co)	1000	0.10		1.0
Chromium (Cr)	1000	0.10		1.0
Iron (Fe)	10000	0.020		2.0
Magnesium (Mg)	10000	0.050		5.0
Manganese (Mn)	1000	0.020		0.20
Molybdenum (Mo)	1000	0.20		2.0
Antimony (Sb)	1000	0.50		5.0
Strontium (Sr)	1000	0.050		0.50
Tin (Sn)	1000	0.50		5.0
Vanadium (V)	1000	0.10		1.0
Zinc (Zn)	1000	0.20		2.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.8.3 Preparation of IDL/MDL Stock C-ICP

Add 20mL to 30mL of reagent water to a clean100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Potassium (K)	10000	1.0	100	100
Sodium (Na)	10000	0.10		10

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.8.4 Preparation of the IDL/MDL Check Solution-ICP

Add 200mL to 300mL of reagent water to a clean 1000mL volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

IDL/MDL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-ICP	10	1000
Stock B-ICP	10	
Stock C-ICP	5.0	

Dilute to a final volume of 1000mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

The IDL/MDL-ICP Check Solution contains the following elements at the given concentrations:

Element	Concentration(mg/L)
Be	0.0010
Mn	0.0020
Ba	0.0040
Sr	0.0050
Ag, Cu, Co, V, Cd, Cr	0.010
Fe, Mo, Zn, Ni	0.020
B	0.025
Pb	0.040
Ca, Mg, Na, Sn, Sb	0.050
Al, As, Se, Tl	0.10
K	0.50

8.9 Preparation of the IDL/MDL-Trace Solution

The IDL/MDL solution is used in this procedure for two purposes:

1) To determine the Instrument Detection Limit (IDL) of each target compound on a quarterly basis; and
2) To determine the Method Detection Limit (MDL) of each target compound on an annual basis.

8.9.1 Preparation of IDL/MDL Stock A-Trace

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Silver (Ag)	1000	0.040	100	0.40
Arsenic (As)	1000	0.20		2.0
Barium (Ba)	1000	0.020		0.20
Beryllium (Be)	500	0.010		0.050
Cadmium (Cd)	1000	0.040		0.40
Lead (Pb)	1000	0.10		1.0
Antimony (Sb)	1000	0.20		2.0
Selenium (Se)	1000	0.20		2.0
Thallium (Tl)	1000	0.20		2.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.9.2 Preparation of IDL/MDL Stock B-Trace

Add 20mL to 30mL of reagent water to a clean 100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Cobalt (Co)	1000	0.030	100	0.30
Chromium (Cr)	1000	0.10		1.0
Copper (Cu)	1000	0.10		1.0
Manganese (Mn)	1000	0.020		0.20
Molybdenum (Mo)	1000	0.040		0.40
Nickel (Ni)	1000	0.10		1.0
Tin (Sn)	1000	0.20		2.0
Vanadium (V)	1000	0.060		0.60
Zinc (Zn)	1000	0.10		1.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.9.3 Preparation of IDL/MDL Stock C-ICP

Add 20mL to 30mL of reagent water to a clean100mL volumetric flask. Add 1mL of concentrated nitric acid (HNO_3) and 5mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Aluminum (Al)	10000	0.20	100	20
Calcium (Ca)	10000	0.10		10
Iron (Fe)	10000	0.10		10
Magnesium (Mg)	10000	0.10		10
Potassium (K)	10000	0.20		20
Sodium (Na)	10000	0.040		4.0

Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

8.9.4 Preparation of the IDL/MDL Check Solution-Trace

Add 200mL to 300mL of reagent water to a clean 1000mL volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. The standard will have an acid concentration of 1% HNO_3 and 5% HCl when diluted to volume. Add the volumes of the stock standards given in the following table to the volumetric flask:

IDL/MDL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-Trace	5.0	1000
Stock B-Trace	5.0	
Stock C-Trace	5.0	

Dilute to a final volume of 1000mL with reagent water. Store the standard at room temperature. Prepare this solution every six months or sooner if needed or required.

The IDL/MDL-Trace Check Solution contains the following elements at the given concentrations:

Element	Concentration(mg/L)
Be	0.00025
Ba, Mn	0.0010
Co	0.0015
Ag, Cd, Mo	0.0020
V	0.0030
Cr, Cu, Ni, Pb, Zn	0.0050
As, Sb, Se, Sn, Tl	0.010
Na	0.020
Ca, Fe, Mg	0.050
Al, K	0.10

8.10 Preparation of the Linearity Check Solutions

The linearity check solutions are prepared individually according to the following equation:

$$Vs = \frac{Vlc \otimes Clc}{Cs}$$

where
Vs = volume of stock standard (mL)
Cs = concentration of stock standard (mg/L)
Vlc = volume of linearity check standard to prepare (mL)
Clc = concentration of linearity check standard to prepare (mg/L)

The linearity check solutions are prepared at the concentrations specified in Table 1. Prepare sufficient volume to perform the linearity check, maintaining the hydrochloric acid concentration at 5% by volume and the nitric acid concentration at 1% by volume.

9.0 SAMPLE PREPARATION

The sample preparation and digestion procedures are listed in the following SOPs:

MATRIX	SL SOP
Aqueous and leachate samples	ME50
Soils and Sediments	ME51
Wastes and oils	ME51

10.0 ANALYSIS PROCEDURE

10.1 Calibration/Standardization of the ICP

10.1.1 Initial Calibration/Standardization

10.1.1.1 The ICP is turned on and allowed to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If optics were turned off allow 2 hours warm up time.

10.1.1.2 Radial plasma ICP: A 1000mg/L Yttrium standard is aspirated into the plasma and the flow of the argon into the plasma is adjusted. The "red flame tongue" inside the plasma should be at the same height as the glass cylinder surrounding the torch at the proper argon flow. The argon flow will be about 0.6 L/minute. As the nebulizer ages, the flow may have to be increased or the nebulizer cleaned/replaced.

10.1.1.3 Mirror adjustment-for ICPs equipped with "D-shaped" mirror. This will entail a horizontal profile of the "D" shaped mirror using the manual profile program maximizing for intensity.

10.1.1.4 Run the "Automatic Profile" program. The "automatic profile" of the instrument should be checked twice a day to compensate for changes in air pressure, humidity, and temperature. If the environment of the instrument is such that daily changes in the instrument profile are extreme, the instrument should be "profiled" every few hours.

10.1.1.5 Analyze the calibration standards and calibrate the ICP. ***If using a multi-point calibration, use the Calibration/Analysis and Curvefit programs to calibrate the instrument.***

10.1.1.6 The highest concentration calibration standard is reanalyzed after the instrument is standardized as an "unknown". The results for the re-analysis of the highest concentration calibration standard must be within +/- 5% of the true value for each target analyte.. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

10.1.1.7 The QC Check standards (ICV) and the Calibration Blank (ICB) are analyzed as a check on the instrument calibration.

10.1.1.7.1 (EPA Method 6010) The results for the target compounds in the initial calibration verification (ICV) must be within the +/- 10 % of the true value.

10.1.1.7.2 (EPA Method 200.7) The results for the target compounds in the initial calibration verification (ICV) must be within the +/-5.0 % of the true value. When performing 200.7 work, note that this solution should be prepared fresh weekly.

10.1.1.7.3 (EPA 6010/200.7) The results for the target compounds in the initial calibration blank (ICB) must be less than the COMPQAP Table 5 Reporting Limit (RL).

10.1.1.8 The RL/PQL Check Solution is analyzed. The results for the analysis of the RL/PQL Check solution are not checked against a set of criteria but is analyzed to demonstrate that the ICP is capable of detecting the target compounds at or near the reporting limit (RL).

10.1.1.9 The ICP Interference Check Sample is analyzed.

10.1.2 Continuing Calibration

10.1.2.1 The calibration of the ICP must be verified every 10 samples by the analysis of the analysis of the QC Check Solutions (CCV) and the Calibration Blank (CCB).

10.1.2.1.1 (EPA Method 6010/200.7-DW) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/- 10 % of the true value.

10.1.2.1.2 (EPA Method 200.7-NPDES) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/-5.0 % of the true value.

10.1.2.1.3 (EPA 6010/200.7) The results for the target compounds in the continuing calibration blank (CCB) must be less than the Reporting Limit (RL).

10.1.2.2 ICP Interference Check Solution and the RL check solution are analyzed at the beginning and end of each analytical sequence.

10.2 Sample Analysis

10.2.1 The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.2.2 The samples are analyzed in a sequence as follows:

INSTRUMENT WARM-UP
ARGON FLOW ADJUSTMENT-1000mg/L Yttrium (Y)
MIRROR ADJUSTMENT
PROFILE
INITIAL CALIBRATION (STANDARDIZATION/*CALIBRATION* OF THE ICP)
REANALYSIS OF HIGH CONCENTRATION CALIBRATION STANDARD AS A SAMPLE
INITIAL CALIBRATION VERIFICATION (ICV)
INITIAL CALIBRATION BLANK (ICB)
DETECTION LIMIT CHECK SOLUTION
ICP INTERFERENCE CHECK SOLUTION A (ICSA)
ICP INTERFERENCE CHECK SOLUTION AB (ICSAB)
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB

The analytical sequence must end with the analysis of the detection limit check standard, ICSA, ICSAB, CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.2.3 Determine the concentration of the samples and QC items using the procedures of Section 11.

10.2.3.1 If the concentration of a sample is above the linear range of the ICP, the sample digestate must be diluted and reanalyzed.

10.2.3.2 The amount of sample digestate needed to prepare the desired dilution is determined form the following equation:

$$V_{digest} = \frac{Vf_{vol}}{DF}$$

where
Vf_{vol} = final volume of diluted sample (mL)
V_{digest} = volume of sample digestate used to make the dilution (mL)

10.2.3.3 The dilution factor is calculated as follows:

$$DF = \frac{Vf_{vol}}{V_{digest}}$$

where
Vf_{vol} = final volume of diluted sample extract (mL)
V_{digest} = volume of sample extract used to make the dilution (mL)

NOTE: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE
A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$DF = \frac{100mL}{1.0mL} = 100$$

Dilutions are prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

10.3 Dilution QC Check

A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

10.3.1 Select a sample digestate that contains one or more target analytes at a concentrations greater than 10X the reporting limit.

10.3.2 Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

10.3.3 Compare the results of the diluted and un-diluted aliquots of sample digestate.

10.3.4 If the results of the dilution are within $\pm$ 10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than than $\pm$ 10%, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike (see section 10.4).

If the concentration of the analyte in the sample is not at least 50 times the instrument detection limit, evaluate the post-digestion spike.

10.4 Post-digestion Spike QC Check
A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. This should be the same sample selected for dilution in 10.3, above.

10.4.1 Transfer 10mL of a digestate to a suitable vial.

10.4.2 Spike the sample with 0.10mL of ICP Matrix Spike I and 0.10mL of ICP Matrix Spike II. The theoretical concentration of the post digestion spike is the same as the LCS or MS if the volume of spiking solution is discounted.

10.4.3 Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed).

10.4.4 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \times 100$$

where

Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = theoretical concentration of spike (ug/L) (See 10.2.5.2)

10.4.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 75-125% recovery.

Result of Post Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<75% recovery but >50% recovery	Analyze all associated samples by single point method of standard addition and quantify by using MSA or qualify all associated samples on report
<50% recovery	Dilute digestate and repeat spike. Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples) If recoveries are not 75-125%, analyze all associated samples by single point MSA. Note – high level of target analytes may inhibit spike recovery. Consult the supervisor in events where high levels of targets appear to be interfering

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high.

10.4.6 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgement when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyzed to determine the extract of the matrix interferences.

10.5 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, Vx, are taken. One aliquot is spiked with a solution of known concentration, Cs. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The concentration of both aliquots are measured and the sample concentration, Cx, is calculated:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

where

S_1 = absorbance or concentration of the spiked aliquot
S_2 = absorbance or concentration of the un-spiked aliquot
Vs = Volume of spike solution

Example:		
	Sample concentration (S_2):	523 ug/L.
	Spike solution concentration (C_s):	50,000 ug/L
	Volume of spike solution (V_s):	0.10mL
	Volume of sample aliquots (V_x):	10mL
	Spiked sample concentration (S_1):	951 ug/L

C_x = [(523)*(0.10)*(50,000)]/[(951-523)*10)] = [2,615,000]/[4280] = 611 ug/L

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Aqueous and Leachate Samples

Aqueous samples are routinely reported in mg/L while the ICP is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

11.1.1 The concentration of the target analyte in liquid samples is calculated as follows:

$$Concentration\ (mg/L) = ug/L(from\ printout)\ x\ \frac{F}{V}\ x\ DF \otimes \frac{1mg}{1000ug}$$

where
F = final volume of the sample digestate (L)-usually 50mL (0.050L)
V = volume of sample digested (L)
DF = dilution factor

11.1.2 The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

$$Concentration(mg/L) = RLqap \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
RLqap = reporting limit from SL QAP (ug/L)
F = final volume of the sample digestate (L)
V= volume of sample digested (L)
DF = dilution factor

The CQAP Reporting Limits assumes:
F = 50mL V = 50mL DF = 1

11.2 Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the ICP is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

11.2.1 The concentration of the target analyte in soil and solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = ug/L(from\ printout) \otimes \frac{F}{W \otimes solids} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
F = final volume of the sample digestate (L)
W = volume of sample digested (kg)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

For example, if the percent solids is 85%, the decimal equivalent is 0.85; if the %solids is 100%, the decimal equivalent is 1.0.

11.2.2 The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = RL_{qap} \otimes \frac{0.0010kg}{W \otimes solids} \otimes \frac{F}{0.100L}\ x\ DF$$

where
RLqap = reporting limit from Table 5, SL COMPQAP
W = weight of sample digested (kg)
F = final volume of the sample digestate (L)
V = volume of sample digested (L)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

The SL COMPQAP Table 5 Reporting Limits assumes

F = 0.100L (100mL)
DF = 1
W = 0.0010kg (1.0g)
solids = 1.0

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 SL SOP AN02: *Analytical Batching*, Table 13.1 of the SL QAP, and the SOP Summary provide guidance on evaluating QC and sample data.

12.2 The method detection limit (MDL) is determined annually in accordance with SL SOP CA90. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP.

12.3 Determination of the Instrument Detection Limit (IDL)

The difference between the MDL and the IDL is the *digestion step*. The MDL samples are prepared and digested prior to analysis. The IDL is defined as three times the standard deviation of seven replicate analyses analyzed over three non-consecutive days. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP.

12.4 For instrument calibration, if any fit, other than linear, is utilized for the calibration of the ICP (i.e., Curvilinear or Full Fit) the upper limit of the linear range is the concentration of the High Standard.

13.0 TROUBLESHOOTING

No items in this revision.

14.0 PREVENTIVE MAINTENANCE

See Section 10 of the current SL QAP.

15.0 REFERENCES

15.1 Savannah Laboratories' ***Comprehensive Quality Assurance Plan***, and *Savannah Laboratories Corporate Quality Assurance Plan*, current revisions.

15.2 ***Methods for Chemical Analysis of Water and Waste***; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.

15.3 ***Test Methods for Evaluating Solid Waste, Third Edition***; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986.

15.4 ***Methods for the Determination of Metals in Environmental Samples***; US EPA Office of Research and Development. Washington, DC.

METHOD SUMMARY - ICP ANALYSIS

HOLD/STORAGE

Container	Minimum 250mL plastic bottle with plastic or Teflon-lined lid
Preservative	HNO3 to pH <2 in the field. If dissolved metals are required, filter the samples before preservation.
Storage	Liquids preserved to pH <2 may be stored at room temperature until preparation. Solid samples must be stored at 4C (less than 6C but not frozen) until preparation.
Hold Time	Samples may be held for up to six months from the time of collection.

SAMPLE PREPARATION

Samples should be prepared with the appropriate matrix-specific procedure.

ANALYTICAL SEQUENCE

Ignite Plasma	Follow instrument manufacturer's guidelines and allow instrument to stabilize for at least 60 minutes.
Check viewing height	If applicable to instrument aspirate 1000 ppm yttrium std. and make sure the yttrium "tongue" is set to be at the top of the outer cylinder of the torch to 1 - 2 mm above the torch.
Check horizontal (mirror) profile	Verify horizontal profile is maximized according to instrument manufacturer's recommended procedure.
Profile Instrument	Record the profile intensity and the caliper reading so that instrument drift can be monitored.
Initial Calibration	Calibrate with a blank and a high standard or a blank and three standards. Verify calibration by reanalyzing high standard.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the run. ICV solution must come from a source other than the calibration standard source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with concentrations at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run Analyze a continuing calibration blank after every CCV.
Interference Check Solutions	At the beginning and the end of an analysis run, verify the inter-element and background corrections by analyzing the interferent check solutions (ICSA & ICSAB).
Detection limit check solution	At the beginning and the end of an analysis run and verify the accuracy at the PQL by analyzing a solution at the SL PQL
Serial Dilution	For new or unusual matrices, perform serial dilution (1/5) on a representative sample.
Post Digestion Spike Recovery.	To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). The post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration in the sample is not at least 50 times the instrument detection limit.

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 std. and 1 blank	
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Highest Standard	Immediately after every calibration	Recoveries within ± 5% of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within ±10% 200.7 = within ± 5%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10% of the true value, 200.7-NPDES - within ± 5% 200.7-Drinking Water – within ±10%	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less that the PQL/CRDL	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning and end of an analysis run	Determined values must be within ± 20% of the true values. Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Lab control sample	One per batch of twenty samples or less	6010B: SL QAP 200.7: 85-115%	Redigest and reanalyze batch
Preparation blank - SW846	One per batch of twenty samples or less	|result| <PQL or result <5% of the analyte level in the sample.	Redigest and reanalyze batch
Preparation blank - 200.7	One per batch of twenty samples or less	|result| <CRDL/PQL or result <10% of the analyte level in the sample	Redigest and reanalyze batch
MS/MSD - SW846	One set per batch of twenty samples or less	SL QAP	Flag and report data
1/5 Dilution	One per batch of twenty samples or less	See section 10.3.4	
Post Digestion Spike	One per batch of twenty samples or less	See section 10.4.5	
Detection Limit Check Solution	At the beginning and end of an analysis run	Detection	Stop the analysis, fix the problem and reanalyze the affected samples.

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L) ICP/Trace	Linear Range Std. Conc. (mg/L)* ICP/Trace	MATRIX SPIKE CONC. (mg/L) Water (mg/L)	Soil (mg/kg)
Aluminum (Al)	308.215	10	1.0/5.0	0.20/0.20	750/800	2.0	200
Antimony (Sb)	206.838	10	1.0/0.50	0.050/0.020	50/10	0.50	50
Arsenic (As)	189.042 193.696	1.0	1.0/0.50	0.10/0.010	50/25	2.0	200
Barium (Ba)	493.409	10	1.0/5.0	0.010/0.010	100/10	2.0	200
Beryllium (Be)	313.042	1.0	1.0/0.50	0.0050/0.0050	30/10	0.050	5.0
Boron (B)	249.678	10	1.0/5.0	0.050	100	1.0	100
Cadmium (Cd)	226.502 228.802	1.0	1.0/5.0	0.0050/0.0050	30/10	0.050	5.0
Calcium (Ca)	317.933 315.887	10	1.0/5.0	0.50/0.50	1000/800	5.0	500
Chromium (Cr)	267.716	10	1.0/5.0	0.010/0.010	100/25	0.20	20
Cobalt (Co)	228.616	1.0	1.0/0.50	0.010/0.010	100/25	0.50	50
Copper (Cu)	324.754	10	1.0/5.0	0.025/0.025	100/50	0.25	25
Iron (Fe)	259.940 271.441	10	1.0/5.0	0.050/0.050	800/800	1.0	100
Lead (Pb)	220.353	1.0	1.0/0.50	0.050/0.0050	100/5	0.50	50
Lithium (Li)							
Magnesium (Mg)	279.079	10	1.0/5.0	0.50/0.50	1000/1000	5.0	500
Manganese (Mn)	257.610	10	1.0/5.0	0.010/0.010	100/50	0.50	50
Molybdenum (Mo)	202.030	1.0	1.0/0.50	0.010/0.010	50/50	0.50	50

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L) ICP/Trace	Linear Range Std. Conc. (mg/L)* ICP/Trace	MATRIX SPIKE CONC. (mg/L) Water (mg/L)	Soil (mg/kg)
Nickel (Ni)	231.604	5.0	1.0/2.5	0.040/0.040	100/10	0.50	50
Phosphorus(P)							
Potassium (K)	766.491	20	10/5.0	1.0/1.0	1000/50	5.0	500
Selenium (Se)	196.026	10	1.0/5.0	0.10/0.010	50/25	2.0	200
Silica (SiO_2)	251.611	10	1.0/5.0	0.50	100	5.0	500
Silver (Ag)	328.068	1.0	1.0/5.0	0.010/0.010	30/5.0	0.050	5.0
Sodium (Na)	588.995	10	1.0/5.0	0.50/0.50	1000/20	5.0	500
Strontium (Sr)	421.552	10	1.0/5.0	0.010	100	0.50	50
Thallium (Tl)	189.042 190.801 377.572	10	1.0/5.0	0.50/0.010	30/30	2.0	200
Tin (Sn)	189.989	10	1.0/5.0	0.050	100/50	1.0	100
Titanium (W)							
Vanadium (V)	292.402	10	1.0/5.0	0.010/0.010	50/50	0.50	50
Zinc (Zn)	213.856	5.0	1.0/2.5	0.020/0.020	100/20	0.50	50

*For guidance only-instrument sensitivity will vary.

MISSION STATEMENT

Triangle Laboratories is in the business of applying scientific knowledge and measurements to the solution of health, environmental and other issues confronting society.

Beliefs

We believe that we must excel in relationships with our customers, our employees, and our investors while establishing leadership in our technology and operations management.

We believe that in all things and at all times our behavior must follow the highest ethical standards. This includes commitments made to customers, suppliers, employees, investors, and to one another.

We believe that to our customers, we must be the laboratory of choice. Our marketing program will always honestly inform. We will set the quality and timeliness standards in our markets. We will structure our company so that we have the flexibility and versatility required to be responsive to customer's needs. We will work until the customer is satisfied.

We believe that for our employees, we must be the employer of choice. Through the application of high ethical standards, maintenance of efficient operations and a respect for diversity, we will provide a work environment that enriches and builds people while giving them an opportunity to excel and enjoy the dignity, pride, and material rewards of being part of a winning team.

We believe that for our investors, we must commit to the development of long term value in their investment. This will be accomplished by taking those risks that have an appropriate probability of reward, controlling expenses to maintain high profitability and aggressively seeking opportunities to achieve growth through expansion of existing business and developing new business opportunities.

We commit ourselves to conducting research and development so that we are always a leader in technology, to apply the knowledge gained to maintain efficient operations and to service our customers needs in a timely manner while providing a reasonable profit for our investors.

Section 1

INTRODUCTION

This manual is a description of the quality assurance program employed at Triangle Laboratories, Inc., referred to hereafter as Triangle Labs. It is intended to provide employees, accrediting agencies, and clients with the information needed to understand how an effective quality assurance system is maintained at Triangle Labs. The QA Manual is divided into fifteen sections and several appendices. The first three sections pertain to the manual itself. Sections 4 - 7 provide general descriptions of Triangle Labs, including its objectives, policies, facilities, organization, personnel, and services. The remaining sections describe specific quality assurance activities as practiced within different functions or work units. The order of sections 8 - 12 closely follows that of the production process at Triangle Labs. The appendices provide supplemental materials that support the descriptions in the QA Manual sections.

Written procedures for implementing the activities described in this manual are maintained as standard operating procedures (SOP's) and as department specific training procedures. The SOP's are made available to the operating staff through the widely distributed SOP Manuals. The training procedures are maintained by the department managers. The provisions of this manual are binding upon all laboratory personnel assigned responsibilities described herein. All laboratory personnel must adhere implicitly to the Standard Operating Procedures.

Section 2

AUTHORIZATION

The quality assurance system described in this Quality Assurance Manual has the absolute support of the management at Triangle Labs as indicated by the signatures below.

The provision of quality analytical services to our customers has given us an enviable reputation and has made us a leader in the industry. Assuring that we maintain this status in providing quality products to our customers is the responsibility of every member of the laboratory staff. It is expected that everyone concerned will use this manual as a guide to quality improvement and to maintenance of our current standing as a quality-oriented laboratory.

Authorization of the QA manual is by:

Signature:	Date:
J. Ronald Hass, Ph. D. President and Chief Executive Officer	09/29/00
Gregory Johnson Quality Assurance Manager	09/28/00
Philip W. Albro, Ph. D. Technical Director	9/28/00

Uncontrolled Copy

TABLE OF CONTENTS

Sections

Appendices

Section 3

MANAGEMENT OF THE QUALITY ASSURANCE MANUAL

The Quality Assurance Department is responsible for the publication and distribution of the Quality Assurance Manual. The manual is submitted to senior management for review and authorization annually. As major changes are implemented in the quality assurance system, the Quality Assurance Manual is revised and submitted to management for authorization. The assistance of laboratory management is essential for the publication of the QA Manual. Department specific information is supplied by the department supervisors for inclusion in the manual.

The authorization signatures found in Section 2 of the manual signify management review and approval of the Quality Assurance Manual. The authorization section must be kept current and reflect any organizational changes affecting the authorizing positions.

Document control procedures are applied to the distribution of the Quality Assurance Manual. Controlled copies are serially numbered and are updated each time a section is revised. Controlled copies of the manual may be distributed to an individual or a department. Uncontrolled copies may be issued to persons or organizations outside of Triangle Labs. These copies are distinctly marked "uncontrolled" and are not subject to updates upon revision of the manual. A distribution list is maintained for all controlled copies of the Quality Assurance Manual.

Upon revision, all text added or changed since the last issue of each section is marked with a vertical bar in the left margin.

Section 4

OBJECTIVE AND POLICIES

Objective

The objective of the staff at Triangle Labs is to provide products and services which satisfy our clients' expectations and definitions of quality and which are legally defensible.

Policies

The management of Triangle Labs supports the following policies in order to achieve the objective and promote the overall quality assurance program:

- Standard operating procedures shall be implemented in order to determine client requirements and to clearly communicate these requirements within the laboratory.
- Organizational emphasis on quality improvement will take place through strong management commitment and leadership, employee empowerment and teamwork.
- A comprehensive quality control system shall be established and maintained in order to verify and assure continued precision and accuracy of analytical results.
- Adequate training on laboratory operations shall be available to all employees whose decisions may affect the quality of laboratory products.
- A comprehensive program of documentation shall be implemented to ensure maintenance of accountability and traceability throughout the analytical process.
- Measures shall be implemented to ensure that sample integrity is protected.
- Validation studies shall be performed for each analytical method, including extensive evaluations whenever major modifications have been implemented.
- The instrumentation, equipment, and materials used in the production process shall be controlled (i.e., purchased, verified, calibrated, maintained, monitored, and evaluated) to ensure that required standards are met.
- A comprehensive program for data reduction, validation, reporting, and archival shall be implemented.

- Preventive and corrective actions shall be taken to eliminate the causes of potential or actual nonconformance. Emphasis shall be placed on preventive measures.
- Measures shall be implemented in order to meet the requirements set forth by agencies from whom certifications and accreditations have been granted.

Section 5

LABORATORY DESCRIPTION

Triangle Laboratories, Inc.

The location, mailing address, and phone numbers for Triangle Laboratories, Inc. are:

Triangle Laboratories, Inc.
801 Capitola Drive
Durham, North Carolina 27713

P.O. Box 13485
Research Triangle Park, North Carolina 27709

(919) 544-5729
(919) 544-2113 (Facsimile)

Triangle Laboratories, Inc. is a privately held subchapter C Corporation registered and incorporated in the state of Delaware. Triangle Laboratories has been in business since 1984 and has established an unparalleled reputation for integrity and quality while undertaking the most challenging work in its industry. The company experienced rapid growth during the emergence of the environmental market. Recognizing the necessity of diversification even while the environmental business was in full swing, the company expanded internationally as well as moving into new markets. Triangle Laboratories currently serves two major market areas, environmental and pharmaceutical.

Facilities and Instrumentation

Triangle Laboratories, Inc. currently occupies more than 50,000 square feet. The facility is divided according to work function, including separate areas for sample receipt; sample preparation, standard, and glassware preparation; sample and data storage; instrumentation; report generation, quality assurance; shipping; maintenance; and business/management offices.

Analytical instrumentation at Triangle Labs includes: high resolution gas chromatograph/high resolution mass spectrometers (HRGC/HRMS); high resolution gas chromatograph/low resolution mass spectrometers (HRGC/LRMS); high pressure liquid chromatograph/mass spectrometer/mass spectrometers (HPLC/MS/MS); high pressure liquid chromatograph (HPLC) with ultraviolet detector (UV); gas chromatographs (GC) with electron capture detectors (ECD) and flame ionization detectors(FID); AOX/TOX adsorption module and microcoulometric titration systems; ion chromatographs (IC).

Well maintained equipment is essential in assuring the timely delivery of complete, high quality analytical data to clients. This is facilitated through a program of regular maintenance for all equipment, equipment redundancy, an ample stock of spare parts, and an inventory of specialized test equipment to support rapid repair when unscheduled maintenance is required. Service technicians are available through contracts with local providers for most of the instruments. Procedures and schedules for preventive maintenance are available in several SOP's. All instrument maintenance, both preventative and corrective, is recorded in the dedicated maintenance logbook assigned to each instrument.

Environmental and Security Systems

Triangle Labs provides a secure environment for our employees, guests, clients, samples and analytical data.

Access Standard procedures require that all exterior doors remain locked via keylock or combination lock unless manned. Visitors are required to sign the Visitor Log and must be accompanied by an employee of Triangle Labs.

The defined high security areas include all laboratories, data archives, computer system, data reduction offices, and quality assurance offices. Entry into these areas of the building are controlled by combination locks on the internal and external entry doors. Visitors must be accompanied by an employee of Triangle Labs at all times inside the high security area.

Several rules apply to protecting the combination lock codes. The combinations are changed periodically. New combinations are supplied to the active employees only by the employee's supervisor or the facility manager. When accompanied by visitors, employees obscure the punch lock combination from view.

Security All doors are locked after hours and require a key for entry.

Archives Limited access archive facilities are maintained that house all Triangle Labs copies of analytical reports, raw data, inactive logbooks, magnetic tapes and other data which facilitate traceability of analytical results. Materials housed in the archives are packaged to reduce potential damage from fire and water.

Chemical Storage and Disposal All chemicals are stored in appropriate cabinets and are properly disposed of when necessary. All flammable solvents are kept in OSHA and NFPA approved cabinets. Acids are stored in OSHA approved acid cabinets. An authorized waste carrier is contracted to pick up lab waste monthly and dispose of it, usually by incineration, meeting all regulatory requirements. Post-analysis disposition of samples is dependent upon client requests. Remaining sample material may be returned to the client, safely discarded, or archived for a specific period of time.

Environmental Control

The working and storage environments are maintained in a safe and appropriate manner. Heating, ventilation and air-conditioning systems satisfy the needs of personnel, equipment and supplies. Lighting, noise and other environmental factors are also considered and kept at appropriate levels. Safety measures which protect personnel and property from injury or illness include the following: fume hoods, fire extinguishers and blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes and spill control kits. Triangle Laboratories has contracts which provide an occupational health program.

Accreditations, Certifications, Licenses and Registrations

Triangle Laboratories, Inc. has received approval from several state and national agencies. The American Association for Laboratory Accreditation has conferred accreditation upon Triangle Labs for technical competence in environmental testing. The laboratory has been validated by the United States Army Corps of Engineers, and while not currently under contract, Triangle Labs has performed organic analyses under the United States Environmental Protection Agency (USEPA) Contract Laboratory Program. Triangle Labs is registered under current Food and Drug Administration (FDA) regulations to engage in the testing of drugs; has received registration under the provisions of the Clinical Laboratory Improvement Amendments of 1988 (CLIA) to perform high complexity testing (dioxin and PCB's) of human samples; has been licensed, and has been provisionally certified by several USEPA regions to analyze drinking water samples for dioxin.

Section 6

ORGANIZATION AND PERSONNEL

Organization

Responsibility and Authority

At Triangle Labs, the management structure is shown in the Organizational Chart in Appendix 1A. Responsibilities and authority of key personnel are summarized later in this section. Brief resumes of key Triangle Labs personnel may be found in the company's Statement of Qualifications.

Verification Resources and Personnel

Verification activities include inspection and monitoring of process and product quality and auditing of the quality system, processes and products. Provision is made for personnel to be trained and have responsibility for these activities.

Production personnel, under the direct supervision of team leaders, are responsible for the inspection and monitoring of in-process and final products. Audits of the laboratory systems and products are performed by personnel independent of those performing the laboratory work. Quality system audits are carried out by Quality Assurance Department personnel, while data audits (audits of the final product) are carried out by employees in both Production and Quality Assurance.

Effective verification activities are achieved by the provision of adequate resources to personnel. These resources include adequate training, time for verification activities, knowledge about requirements, documented procedures, access to quality records, and adequate supplies and equipment necessary to perform verification.

Management Representative for Quality Assurance

The Quality Assurance Manager reports directly to the President, functions independently of production, and has the authority to implement and maintain the quality system. The management of Triangle Labs presents a strong commitment towards the important role of quality assurance in its organization. The Quality Assurance Manager and other members of the Quality Assurance Department interact frequently with personnel at all levels throughout the organization.

Management Review

A formal management review of the quality system occurs annually. The purpose of this review is to ensure that the quality system remains effective, meets the quality objectives and policies stated in Section 4 of this manual, and satisfies the requirements of state, national, and international certifications held by Triangle Labs. Records of management reviews shall be maintained in the Quality Assurance Department.

Personnel

Job Descriptions of Key Technical Personnel

While not all-inclusive of assigned duties, the following are brief descriptions of the chief technical personnel at Triangle Labs.

President/Chief Executive Officer: management of administrative, business, quality assurance, personnel and production activities; direct supervision of the Production Manager, the Quality Assurance Manager; and the Technical Director, minimum qualifications - education: Ph.D. Chemistry, experience: 10 years analytical chemistry.

Quality Assurance Manager: coordination and management of the Quality Assurance Department; reports directly to the President; responsible for overseeing all quality aspects of the laboratory; specific elements to be maintained are: the Standard Operating Procedures, Quality Assurance Manual; coordination of internal and external audits, performance samples and laboratory certification data; minimum qualifications - education: B.S. Chemistry or equivalent, experience: 5 years in scientific field.

Technical Director: consultation and guidance on specific technical and scientific questions and issues; performs audits of the technical aspects of program operations; reports directly to the President; minimum qualifications - education: Ph.D. Chemistry, experience: 5 years analytical chemistry.

Production Manager: The production manager is responsible for developing production plans to meet commitments made to clients, identifying and resolving issues which impede success, and promptly reporting to the president any issues which cannot be resolved with available resources; minimum qualifications - education: B.S. Chemistry or equivalent, experience: 5 years in scientific field.

Team Leaders: management of a defined production area, instrumentation, reporting and/or sample preparation; minimum qualifications - education: B.S. Physical Science, experience: 2 years general analytical chemistry course or lab experience.

Recruitment Policy

The Human Resources Department of Triangle Labs uses several methods of recruitment. Current employees are offered the earliest opportunity to apply for openings within the facility by posting available positions on the bulletin boards before outside sources are considered for candidates. Then, announcements are made in local newspapers, placement agencies (temporary and permanent), colleges and thè Employment Security Commission offices. The recruitment process consists of collecting applications and resumes, distributing them to the appropriate supervisors, scheduling interviews as requested by supervisors and having candidates meet with relevant staff, a representative from the Human Resources Department and senior Management. The references of promising candidates are investigated prior to making job offers.

Training

Training is provided for new employees and as continuing education for veteran employees, both at the Triangle Labs facility and off-site.

On-Site Training: Training goes on at different levels throughout the facilities. Numerous manuals, texts, videos, SOP's, journals, analytical protocols and in-house instructors are available to trainees. On-the-job training related directly to the position is done by team leaders or other qualified staff. Typically, a trainee goes through a stepwise method to learn procedures pertaining to such areas as analytical methodology, report generation or quality assurance activities: the trainee is given an SOP to read, the trainee observes the trainer performing the procedure, the trainee assists the trainer in performing the procedure several times, the trainee performs the procedure without assistance but with the trainer's frequent inspection of his work, and finally, the individual may perform the procedure without supervision. The Quality Assurance Manual is available to all employees whose activities have a direct impact on product quality. Cross training, supervisory training and other related training takes place on a scheduled basis and is documented for training files.

Off-Site Training: This type of training takes place on an as-needed basis. Recommendations and suggestions about promising educational programs come from all levels of staff. Completed studies are documented and updated regularly in the training files. Courses may be taken at local colleges and universities. Workshops and seminars are often made available by instrument manufacturers, software companies and national associations specializing in analytical chemistry or laboratory quality assurance.

Training Records Maintenance

Résumés, education and experience records, job descriptions and training records are maintained by the personnel department: Résumés are put in a uniform format upon hire. These résumés are updated on an annual basis or as needed. Additional education and experience is updated with the résumés. There is a job description for each position existing within the company. Active training records are kept on file in the work areas. Employees are responsible for maintaining their own training records. These training files contain records for any pertinent on- or off-site educational experiences, orientation records, SOP competence records or self help courses.

Safety and Health Policies

All personnel undertake a two day orientation upon initial employment and on-the-job intensive training concerning health and safety issues. Triangle Labs complies with the OSHA requirement that safety and health training takes place on an annual basis, with a careful introduction to new principles. We have contracted with Concentra to provide us with recommendations for the improvement of the safety and health practices at Triangle Labs. Triangle Labs' policy with respect to health and safety issues is presented in detail in several documents, which are provided to employees.

Section 7

ANALYTICAL SERVICES

Triangle Labs has assembled an international staff of unparalleled expertise in analytical sciences with particular specialization in mass spectrometry and the analysis of complex biological matrices. The skills of the staff are routinely applied to environmental samples, including of air, water, solid and tissue matrices, and to biological samples associated with studies supporting the research efforts of the pharmaceutical industry.

Pharmaceutical Services

Triangle Labs serves the research pharmaceutical industry by providing analytical results for drugs of interest in a variety of biomatrices. This work is typically associated with pharmacokinetic Phase I through Phase IV studies for reporting to the Food and Drug Administration (FDA).

GC/MS and LC/MS/MS methods are typically employed for these analyses. High resolution mass spectrometers and alternate ionization methods are frequently utilized to achieve low detection limits. The staff is also experienced in assays, both GC and LC based, for chiral compounds.

Environmental Services

Triangle Labs provides environmental analytical services which include the preparation and analysis of a wide variety of sample matrices for such analytical categories as:

Volatile and Semivolatile Organic Compounds, including Polychlorinated Biphenyls, by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry

Polychlorinated Dibenzo-*p*-Dioxins, Polychloro-dibenzofurans, Polychlorinated Biphenyls, and Polynuclear Aromatic Hydrocarbons by High Resolution Gas Chromatography/ High Resolution Mass Spectrometry

Polychlorinated Dibenzo-*p*-Dioxins and Polychloro-dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry

Adsorbable Organic Halides and Total Organic Halides by Adsorption and Microcoulometric Titration

Triangle Labs is experienced in the analysis of many matrices, including air, aqueous, plant and animal tissues, soils, and other solids. Air matrices currently analyzed include Modified Method 5 (MM5) samples and Volatile Organic Sampling Trains (VOST). Several auxiliary services are also offered, such as the provision and preparation of sampling containers (e.g., XAD traps, VOST tubes, and bottles).

Contract Review

For all analytical services to be provided by Triangle Labs, contract review is accomplished through the generation of a written quote or contract. Written quotes are utilized for short-term contracts, usually consisting of one analytical project. Written contracts are utilized for long-term contracts consisting of multiple analytical projects. Sales and Client Services personnel are responsible for implementing and documenting contract review. Client requirements, including special needs that are not normally provided by Triangle Labs, are defined and documented in the written quote or contract. Project scientists, who each have expertise in specific analytical services, are consulted to ensure special requirements can be met by the laboratory. If it is decided that the special requirements cannot be met, this is discussed with the client, and a counterproposal may be offered. Information about the capacity of the lab is made available to Sales and Client Services personnel on a regular basis. This practice allows the sales staff to make informed decisions regarding contracted delivery times.

Subcontracted Analyses

In dealing with any analyses that Triangle Labs cannot perform, there are established procedures for subcontracting. Depending on the nature of the client's requests for analyses, two courses of action may be followed. The client may be referred directly to another laboratory, or work may be subcontracted by Triangle Labs to another laboratory. The latter usually takes place at client request. When the subcontracted analysis is one that Triangle Labs has been certified to perform, the subcontract lab must have a quality assurance system in place that is consistent with Triangle's system. Incoming samples which will be subcontracted are subjected to normal sample receipt procedures by the sample custodian. The samples are prepared and shipped to the subcontract laboratory. Results are received at Triangle Labs, a copy is sent to the client, and the original is archived. Triangle Labs invoices the client for the subcontracted work.

Section 8

LABORATORY MATERIALS—PURCHASING AND HANDLING

Purchasing, Receiving, Inspection, Inventory and Storage of Laboratory Materials

Practices utilized for the purchase, receipt, inspection, inventory, and storage of laboratory materials are described in several SOP's. A completed purchase requisition form provides a clear description of the product ordered. This includes, where applicable, a precise identification and reference to any specifications that must be met. Purchases are pre-approved by department heads. The purchasing department orders the material, from an approved supplier whenever possible. Upon receipt of the goods, receiving personnel examine them for damage before signing the bill of lading. Within two days, items and quantities in all shipments are compared with what was ordered and this information is communicated to purchasing and accounts payable. All stocked items are stored in the warehouse and a monthly inventory is performed. Non-stocked inventory is forwarded to the requisitioning person. Reagent materials are assigned expiration dates and placed on shelves so that the older materials will be used first.

Sample Container Cleaning, Storage, Preparation and Shipping

While Triangle Labs does not perform sampling, sampling kits may be provided upon client request. The vials, jars, and bottles contained in the kits are purchased and must be QC class, precleaned, with a certificate of analysis. The certificates of analysis are maintained by Triangle Labs. Since kits are assembled only upon clients' requests, no "ready for shipping" kits are stored. Precleaned glassware is stored in small quantities in house. Sampling materials, such as XAD traps, PUFs and VOST tubes, are also provided to or owned by the client. These are prepared, stored and handled as detailed in several SOP's.

Prior to shipping, glass containers are wrapped in sheets of bubble wrap to prevent breakage. The containers are placed in plastic coolers with non-frozen ice packs and Chain-of-Custody forms, seals and labels enclosed in a ziplock bag. The kit is filled with additional packing material and sealed with tape for shipping.

Glassware Cleaning

All glassware used for the preparation of samples is cleaned as described in written standard operating procedures. These procedures include pre-rinses and soapy water washes. The pre-rinse may be solvent, water or acid solution depending on the analysis for which the glassware will be used. Basins and brushes are kept segregated so that cross contamination is minimized. Glassware used for high concentration analyses is kept segregated from glassware used for low concentration analyses, as is the glassware used for volatile and extractable organic compound. Glassware used for the analysis of extractable organic compounds, including dioxins and furans, is subjected to a solvent soak and rinses with several solvents. All clean glassware is covered with aluminum foil and transferred to a proper storage location, taking care that the glassware is not intermixed with other types of glassware.

Vendor Qualification

Vendors subject to qualification are those who provide critical laboratory supplies, chemicals, and calibration services which directly impact on the quality of our product. Placement on the approved vendor list is based on the vendor's ability to meet one or more qualification factors which cover the purchased product. These factors include but are not limited to:

1. the vendor's quality system or product meets an applicable state, national, or international standard, based on third party certification
2. an acceptable quality assurance plan/survey, or on-site audit;
3. the vendor provides quality inspection documentation with each shipment or batch lot of product;
4. the vendor passes comprehensive inspections of three consecutive product shipments;
5. a demonstrated history of acceptable product supply.

A vendor may be provisionally approved until qualification factor(s) are met, but in-house inspection of each batch lot of material is required. Previously approved vendors may be disqualified due to unacceptable performance.

Client Verification

When required by contract, the client or a representative may verify that purchased products conform to contract specifications. This verification may take place at the vendor's premises or at Triangle Labs. Client verification shall not be used as evidence of effective control of quality by the vendor and shall not absolve Triangle Labs of responsibility to provide an acceptable product.

Section 9

ANALYTICAL STANDARDS

During the analytical process, it is possible to obtain a variety of measurements. These include such measurements as volume, weight, concentration, pH, and temperature, to name just a few. The laboratory must implement practices that facilitate the traceability of these measurements to recognized standards of measurement.

Chemical Standards

The procurement, preparation, handling and storage of chemical standards is critical to the analytical process. It is through these chemical standards that reported analyte measurements in samples are traceable to reference values. Only the highest quality chemicals are used as reference materials at Triangle Labs. Whenever possible, standard solutions will be traceable to national standards, such as NIST, EPA or A2LA certified reference materials. Numerous written procedures describe the management of these analytical standards. These procedures are written to ensure consistency with the requirements of analytical methods and current certifications and accreditations.

Sources of Standards, Traceability and Verification

Triangle Laboratories purchases standards from approved suppliers of chemical standards. Occasionally, clients supply standards specifically for use in the preparation and analysis of their samples. Prior to using these standards, an agreement must be reached with the client about the handling and disposition of their standards. Information about these standards and any client requirements are recorded in the pertinent standards logbook. The chemist receiving a chemical standard shipment verifies that the information on the standard label is consistent with that on the supplier paperwork. Information about the standard is recorded in a standards logbook. Traceability of standard solutions is facilitated by the use of codes that unambiguously identify the supplier, materials and all derived preparations. Non-certified standard materials are verified against certified reference standards, when the latter are available.

Types of Standards

Analytical methodologies define a variety of standard solutions which are used by the laboratory. Included among them are: surrogate spikes, matrix spikes, internal standards, QC check standards, recovery standards, and calibration solutions. The composition and concentration of these solutions must conform to method specifications.

Standards are categorized at Triangle Labs according to the following definitions:

Primary Standard A neat standard received from a supplier.

Stock Standard	A solution of a primary standard at a high concentration, used to prepare secondary standards. These may be prepared in-house or received from a supplier.
Secondary Standard	A solution of one or more stock standards, with each analyte prepared at a selected concentration, to be used as a beginning mixture for preparation of calibration or spike solutions. These may be prepared in-house or received from a supplier.
Working Standard	A solution that will be used without dilution for instrument calibration or sample fortification. These may be prepared in-house from secondary standards, or purchased from a supplier,

Preparation of Standards

The preparation of any standard solution is performed by an experienced chemist, and is documented in the appropriate standards logbook. New standard solutions are prepared as needed. The manner of preparation for a standard solution depends upon the required amount and concentration and its intended application. Several SOPs are utilized to assure the correct preparation and documentation of standard solutions.

All standards are assigned an expiration date. The supplier's assigned expiration date, if provided, is used for neat or primary standards. Otherwise, the expiration date is assigned based upon the supplier's date of preparation and the known stability of the analyte. (Some analytes are known to be highly volatile or to easily degrade or react.) When applicable, assigned expiration dates meet the requirements of analytical methods. A standard mixture is assigned an expiration date no later than that of the oldest components. The expiration date is only a guideline. Standards are removed from production prior to the assigned expiration date if deterioration is observed visually or analytically or if the integrity of the material can no longer be assured.

Analyte or standard components common to calibration solutions and associated sample fortification solutions may be of the same primary source or an independent source. Some methodologies require that primary standards of the same supplier batch or lot number be used for both. Certain spiked QC samples must be prepared from reference material that is independent of the associated calibration standards. New standards are prepared as necessary to meet these requirements.

Inventory and Storage

Documentation for all standards is carefully recorded in relevant standards logbooks and/or computer inventory system. The manner of storage for a standard is determined by its type and expiration date or shelf life. All light sensitive standards are stored in amber vials or bottles. Environmental organic standards are kept in designated refrigerators/freezers. Pharmaceutical standards are stored according to the conditions specified in the associated protocol, validation report or stability report. Analytical standards are never stored together with samples or extracts.

Measurement Equipment

All equipment used for measurement and testing shall meet the specific requirements of pertinent analytical methods and applicable certification agencies. This includes small equipment, such as thermometers, analytical balances, pH meters, autopipetors, and volumetric glassware; as well as large equipment, such as gas chromatographs and mass spectrometers.

Written procedures for the operation of measurement equipment, large or small, shall contain the information described below, where applicable. In addition, Section 11 on "Instrumental Analysis" of this manual contains more specific information about the calibration and operation of large measurement equipment.

- What equipment the procedure is to be performed on, including equipment type
- How the equipment is to be calibrated and used for measurement
- What measurements are to be made
- Acceptance criteria for the calibrations, including the accuracy and precision required
- Corrective action for failed acceptance criteria, including assessment of previous calibration results
- Basis used for calibration (e.g., national standards of measurement, such as NIST, ASTM, and A2LA; participation in EPA and state performance evaluations; round-robin studies with other laboratories)
- Frequency at which the equipment will be calibrated, adjusted and checked
- What records will be maintained to document the calibration and use of measurement equipment

- How the calibration status for equipment is determined (e.g., a sticker or logbook entry)
- What environmental conditions are necessary before measurement equipment may be calibrated or used for measurement
- What adjustments to measurement equipment, including software, cannot be made due to possible invalidation of the calibration setting
- How measurement equipment is to be handled, preserved, and stored in order to maintain accuracy and fitness for use

Section 10

SAMPLE RECEIPT, HANDLING AND PREPARATION

Sample Receipt and Chain-of-Custody

The Sample Custodian or a designated assistant receives deliveries of all samples. A unique project number is assigned to each shipment of samples received from a client, and the first in-house records for the new project, including an internal Chain-of-Custody, are initiated. When samples are hand delivered by a customer, the individual's name is recorded on the internal Chain-of-Custody. The shipping containers, their contents, and accompanying client documentation are examined by the Sample Custodian. Information about the presence and condition of custody seals and the state of preservation of the samples is noted on the internal Chain-of-Custody. Any discrepancies in documentation or problems with sample condition are also noted and brought to the attention of the client, who may provide clarification or further instructions. The Sample Custodian assigns an internal sample ID to each sample, which is labeled on the sample container. The following information pertinent to each sample is recorded on the internal Chain-of-Custody: internal sample ID, client sample ID, sample matrix and storage location. The original internal Chain-of-Custody is placed in storage with the samples. The sample receipt and handling SOP's describe procedures for sample receipt and log-in, chain-of custody, along with those for handling sample shipment containers provided by clients.

Sample Preservation and Security

Samples are stored in a manner which ensures their integrity and security. Samples are stored at temperatures which meet specifications of the methodology and client. Depending on the nature of the sample and the requirements of the method, samples may be stored in a freezer at -70° ± 20° C or at -20° ± 10° C, in a refrigerator or cooler at 4° ± 2° C, or in a cabinet at room temperature. Required preservation techniques may be found in Appendix 4 for most methods employed at Triangle Labs. Quality Assurance Project Plans (QAPP's) and protocols often give specific preservation requirements that must be observed. Addition of chemical preservative to sample containers normally takes place at the time of sample collection. Sample storage facilities at Triangle Labs are located within laboratory areas which are secured by locked doors. Internal chain-of-custody procedures and documentation pertaining to sample possession, removal from storage and transfer are outlined in written procedures. Care is taken to ensure that cross-contamination does not occur during sample storage. Temperatures of cold storage areas are monitored and recorded at least twice a day, and corrective action is taken as necessary. Walk-in coolers housing environmental samples and freezers used for pharmaceutical samples and standards are monitored electronically 24 hours a day. Further details about sample storage and preservation may be found in the sample receipt and handling SOP's.

Sample Preparation Procedures

Samples are prepared in a way that is method and matrix specific. Most environmental samples must be prepared within a method-specified time after sampling. These preparation holding times are complied with to the extent possible. Samples are occasionally received near or beyond the expiration of these holding times. For most methods employed at Triangle Labs, holding times may be found in Appendix 5. Applicable Quality Assurance Project Plans (QAPP's) and protocols must be consulted for project-specific holding time requirements. Many primary extracts require clean-up procedures before they may be injected into a GC or GC/MS analytical system. All sample preparation procedures employed at Triangle Labs are covered by appropriate SOP's.

Sample, Extract, and Digestate Archival and Disposal

The Sample Custodian and other authorized personnel are responsible for the archiving and disposal of raw samples, extracts, and digestates. Raw and prepared samples may not to be archived or disposed of until all of the designated analyses are complete and resultant analytical data are sent to clients. Samples in cold storage are retained there until at least 30 days after data ships. Archive samples are placed in boxes, labeled with the project numbers, and retained in a secured sample archive area for a specific length of time, prior to disposal. Written procedures describe routine archival and disposal practices. Clients are informed about these procedures and are given an opportunity to request exceptions to these routine practices. There is a storage fee for the retention of samples in cold storage or archive longer than the time established by routine practices. The client will be contacted prior to the issuance of this fee.

Sample Return to the Client

When a client has requested the return of samples, the Sample Custodian prepares and ships the samples according to written procedures. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the control of Triangle Labs. Clients are informed that a commercial carrier will deliver their samples.

Sample Loss, Damage, or Unsuitability

It is possible for samples or sample containers to be lost, damaged or determined to be unsuitable, for whatever reason, after initial receipt at Triangle Labs. Whenever this happens, the event is recorded in the sample handling documentation by the observer. The problem is brought to the attention of a Project Scientist, who reports it to the client. Plans for disposition of the affected sample(s) or containers are agreed upon with the client, carried out, and recorded in the project records.

Section 11

INSTRUMENTATION AND EQUIPMENT

Instrumental analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations and other instrument performance tests, analyzing prepared samples, and collecting data from the analyses. Instrumental analysis procedures, frequencies and acceptance criteria are described in several SOP's. A description of data collection and reduction at Triangle Labs is given in Section 12.

Instrument Operating Conditions

The published analytical methods normally define the instrument operating conditions (e.g., temperature programs, column conditions, flow rates). Where applicable, these guideline will be followed. However, they may be modified, for improved performance.

Calibration Procedures and Frequencies

Equipment used for inspection, measuring and testing must meet all specific requirements for proper measurement capability as identified in the pertinent analytical method and applicable certification agency. This includes small equipment and instruments as well as large analytical instruments such as gas chromatographs and mass spectrometers. Calibration procedures and frequencies specific to types of equipment are briefly described below.

The instrumental performance requirements of the published methods will be followed unless otherwise specified for a project. Other performance tests may also be executed to further demonstrate proper functioning of instrumentation.

Small equipment

Thermometers Laboratory thermometers are routinely checked for accuracy against certified, NIST-traceable thermometers. These calibrations are performed annually for mercury or alcohol in glass thermometers, and quarterly for metal thermometers. Infrared thermometer calibrations are verified daily. Correction factors derived from the annual and quarterly calibrations are applied to temperature readings where applicable. NIST-traceable thermometers are professionally calibrated and re-certified annually.

Balances Calibration checks are performed for each day of use for each balance. The calibration consists of a minimum of two weights which encompass the weight the balance will be used to measure. Calibration weight measurements must meet the acceptance criteria listed in the associated balance calibration log book. Each balance is serviced and calibrated by a certified professional, semiannually. The accuracy of the calibration weights are verified annually.

Uncontrolled Copy

Volumetric Glassware All volumetric glassware used at Triangle Laboratories, Inc. must be type "class A." Volumetric glassware is never heated or placed in an oven.

Automatic Pipettes Delivery volumes for the automatic pipettes are checked gravimetrically monthly. Each pipette is checked throughout the volume range of use. Acceptance criteria for continued use is 2% RSD and 97.5 - 102.5% accuracy. Pipettes which fail to meet these criteria are tagged and removed from service until repaired.

pH Meters pH meters are calibrated prior to use each day. The meter is calibrated using a single buffer solution at mid-range and the pH of two other solutions (at low and high range) is measured and recorded to verify the accuracy over the range of the meter.

Conductivity Meters A five point calibration curve using potassium chloride (KCl) solutions is analyzed annually. A single KCl standard solution is used as a check standard each day the meter is used. Acceptance criteria is ±20% of the true value.

Gas Chromatography/Mass Spectrometry (GC/MS) and Liquid Chromatography/Mass Spectrometry (LC/MS/MS)

Tuning and Mass Calibration For high resolution, selected ion monitoring analyses, the high resolution mass spectrometer is tuned to give the required static resolving power, which is checked visually, using an oscilloscope. This measurement is confirmed by the use of a data system. The instrument is then mass calibrated using perfluorokerosene (PFK) or perfluorotributylamine (PFTBA). Mass calibration is adjusted automatically by the data system, to within ± 5 parts-per-million (ppm), approximately once per second during the course of all quantitative analyses.

The mass calibration of a quadrupole mass spectrometer is checked daily through the use of the perfluorotributylamine reference compound (FC-43/PFTBA). The instrument is adjusted to give specified peak ratios for this compound, consistent with the type of analysis to be performed. The GC/MS is tuned prior to performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. For volatiles analyses, 50 ng of bromofluorobenzene (BFB) is used, and for semivolatiles analyses, 50 ng of decafluorotriphenylphosphine (DFTPP) is used.

Initial Calibration For environmental samples, the mass spectrometer response is typically calibrated by analyzing a set of five or more initial calibration solutions, as appropriate for each GC/MS method. Typically each solution is analyzed once, unless the method requires multiple analyses. The relative response factor for each analyte (target compounds, surrogate / internal / alternate standards) is calculated using the expression in Formula 11-1. The mean relative response factor for each analyte is then obtained using the expression in Formula 11-2. Integrated ion currents are utilized for these expressions. An acceptable calibration must meet the method specified criteria for percent relative standard deviations (% RSD) of the mean relative response factors, calculated for each analyte. Failure to meet the criteria will result in corrective action (e.g., locating the source of the problem and adjusting the instrument tuning parameters) before repeating the rejected analysis. Triangle Labs does not analyze any samples unless the performance criteria for calibrations are satisfied.

For pharmaceutical samples, the calibration curve normally consists of a minimum of five standard concentrations analyzed at the beginning and end of the analytical sequence, or are dispersed throughout the analytical run depending on the client's requirements. All standards are used for the regression, with exclusion criteria defined in each method SOP.

Continuing Calibration

For environmental analyses, the initial calibration is verified through the analysis of a continuing calibration standard every 12 hours. The concentration of continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated using the expression in Figure 11-3. An acceptable continuing calibration run must have measured percent differences for the analytes within method specified ranges. Should any criteria for an acceptable calibration not be met, either instrument maintenance is performed such that a new continuing calibration analysis meets all criteria or a new initial calibration will be established before any samples can be analyzed. No samples may be analyzed unless acceptance criteria have been met.

For pharmaceutical analyses, the calibration is verified through the analysis of quality control samples which are interspersed throughout the analytical sequence. The quality control samples are matrix spikes which contain known levels of analyte and are extracted with the samples.

Formula 11-1

$$RRF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where

RRF = *the relative response factor for the analyte*

A_{is} = *integrated area or ion current of the internal standard*

A_s = *integrated area or ion current of the analyte*

C_{is} = *amount of the internal standard*

C_s = *amount of the analyte*

Formula 11-2

$$\overline{RRF} = \frac{1}{n}\sum_{i}^{n}\frac{A_s C_{is}}{A_{is} C_s}$$

where: $\overline{RRF}$ = the mean value of the relative response factors for the analyte

n = the total number of data points derived from the initial calibration

A_{is}, A_s, C_{is} and C_s have the same meaning as in formula 11-1.

Formula 11-3

$$\%D = \frac{RRF_{cc} - \overline{RRF}}{\overline{RRF}} \times 100$$

where:

$\overline{RRF}$ = mean relative response factor for the analyte in the initial calibration

RRF_{cc} = relative response factor for the analyte from the continuing calibration

Ion Chromatography (IC)

The ion chromatograph is typically calibrated by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. Procedures for verifying the calibration curve are method specific.

AOX/TOX Instrumentation

Instrumentation for the determination of AOX/TOX consists of a column adsorption module, titration cell and combustion/microcoulometric system. Several system performance tests are conducted and must meet acceptance criteria prior to sample analysis. The following performance tests are typically conducted, with slight variations between the different analytical methods. Granular activated carbon utilized in the column adsorption module is tested for purity. The titration cell is tested and adjusted based on the results of an injection of sodium chloride solution. Calibration of the combustion/microcoulometric system is accomplished through the analysis of 2,4,6-trichlorophenol. Verification of system performance and calibration is performed during sample testing according to specifications in the analytical methods.

Sample Analysis Procedures

Techniques for quantitative analysis of samples are specific to the analytical methods and sample matrices. Samples may either be subjected to a series of preparation steps prior to instrumental analysis, or they may be ready for analysis upon arrival at Triangle Labs. Most samples must be analyzed within a defined period of time following their collection, receipt at the lab and/or preparation. These analysis holding times are complied with to the extent possible (samples are occasionally received near or beyond the expiration date of holding time). Holding times for most methods employed at Triangle Labs may be found in Appendix 4.

After sample analysis is completed and the data is processed, the analyst reviews the resultant data. If established acceptance criteria are not met, corrective action is taken to resolve problems. Once all the samples in a project have been analyzed and the data have met the criteria, the project documentation (instructions, raw data, reports, etc.) is sent to the next stage for preparation of the final report.

In the event of a discovery of the existence of defective measuring or test equipment which had been used to report data, it must be reported immediately to the area supervisor and all Project Scientists who's projects are affected. The Project Scientist must notify the client both verbally and in writing. If it is a recurring problem which indicates a repetitive failure of the analytical system, it must be reported to the Production Manager, Technical Director and Quality Assurance Manager. A corrective action report is initiated. (See section 14 of this manual for more details on corrective action reports.)

Section 12

DATA HANDLING AND SOFTWARE MANAGEMENT

Data Collection and Reduction

Quality assurance principles are applied in the acquisition of raw data related to chemical measurements. Raw data is "primary data" which will be used to generate "secondary" data (the final analytical report). Data can be acquired manually or electronically. Manually acquired data is hand written on data sheets and in logbooks. Electronically acquired data is acquired from an instrument and instrument/computer interface. Specific definitions and data requirements are detailed in the Raw Data SOP.

Manually Acquired Data Manually acquired data: is recorded on data sheets or in notebooks. The data must be recorded immediately by the analyst in permanent ink. Each entry must be signed and dated immediately after entry. Corrections must not obscure any original entries. Corrections are made by canceling with one line through the original. Each correction must be dated and initialed by the person who made the correction and a reason for the correction must be stated. Data sheets are standardized, preprinted forms which are subject to document control. Data sheets may be bound into a book or may be used as loose sheets depending on the application. Notebooks are bound, consecutively numbered, and subject to a controlled distribution and archival system.

Electronic Data Electronically produced data may consist of chromatograms, spectra, data printouts, and raw quantitation reports. The first accepted hard copy report constitutes the raw data for each sample and calibration. Acceptance is signified by the dated signature of the analyst. The accepted hard copy report must contain the full sample ID or calibration name, file name, as well as date and time of acquisition. Any changes to the raw data hard copies and computer files must be fully documented and clearly attributable to the person making such alterations (e.g., manual integrations are hard-copied for inclusion in the raw data file, with area changes fully documented on the data printouts). No ambiguity in data system printouts as to what peak on a chromatogram corresponds to an analyte of interest is allowed. Computer-collected data is reduced to hard copy as soon as possible. The signed and dated hard-copies of the data files are retained in the project file and are maintained for a minimum of 10 years. The electronic files are safeguarded by a system of disk storage and backup disks to protect loss of data and programs.

There are several different means of data collection, review and reduction, which are dependent upon specific methodology and instrumentation. Data review and reduction of pharmaceutical data normally consists of data acquisition via a dedicated computer with further reduction and data reporting utilizing validated spreadsheets. Regression and sample calculations are verified independently for each pharmaceutical data set.

Data review and reduction of environmental analyses normally follow the guidelines of relevant EPA reference methods to the extent possible. For HRGC/HRMS analyses, established procedures consist of data acquisition and reduction on a Digital Micro VAX and VAX 3100 and further reduction and data reporting using dBase software on a PC. For HRGC/LRMS analyses, established procedures consist of data acquisition and reduction using PC-based software followed by further data reduction and reporting using dBase software. For HRGC analyses, established procedures consist of data acquisition and reduction using PC-based software followed by further data reduction and reporting using dBase software. For AOX/TOX analyses, manual data acquisition from instrument panel readings is followed by data reduction and reporting using spreadsheet software.

All GC/MS data go through several levels of review and inspection, starting with an initial examination in the Instrumentation area, followed by a thorough review before preparation of the report. After preparation of a report, an independent review is performed by a chemist other than the one who prepared the report. At each stage of the analytical process, data are reviewed for completeness, adherence to protocol requirements, and credibility. Results are fully validated, possible compromises of data quality are evaluated, and deviations from protocol requirements are documented. To the greatest extent possible, computer programs are utilized for data reduction. Where manual data procedures are required, data review is performed according to standard operating procedures. This ensures that the results are as independent of the chemist performing the duties as possible. Corrective actions are implemented at the earliest possible opportunity.

Data Validation

The tests performed by Triangle Labs typically involve the performance of complex chemical analyses by a number of chemists. For this reason data validation and coordination are very important. At the conclusion of the analyses, data are checked against the original shipping information and analytical request to be sure that the required analyses have been performed on all samples.

The validity of the data are verified through the analysis of blank samples, duplicate samples and laboratory control or matrix spikes. The blank sample results demonstrate the absence of laboratory contamination of the samples. Duplicate analyses give a measure of analytical precision. The analysis of spike samples permits a measure of accuracy. Data for these QC samples are reviewed as soon as possible after analysis. For example, in the GC/MS area, a data quality checklist is used by the instrument operator at the time of analysis, to verify that all calibration verifications are within tolerance, and that other QC indicators such as spike recoveries and blanks. are acceptable for a project.

Data Reporting

The data are reported as components identified and the quantities present. The final report includes example calculations and descriptions of the equipment and procedures utilized. Complete data packages of all raw sample and calibration data are prepared and archived. These are furnished to the client upon request. Sample flagging procedures for HRGC/HRMS analyses are summarized in the final report.. While sample flagging is not done directly on most HRGC/LRMS analytical reports, problematic results are discussed in the case narrative which accompanies each data package.

Data Package Delivery

Data packages are prepared for delivery by the Shipping and Archive department according to their SOP's. Unless otherwise requested by the client, a copy of the data package is shipped, while the original is retained in a secured archive facility. Reports are fully paginated prior to copying. The data packages are packed to meet the requirements of the commercial carrier chosen for delivery. Packages are delivered by a commercial carrier whose procedures for protecting the data packages are not within the control of Triangle Labs. Should the shipped data package be lost or damaged during delivery, a copy can be quickly prepared as a replacement. Clients are made aware that a commercial carrier will deliver their data packages.

Corrections and Additions to Documentation

The policy for handling additions/corrections of reports already issued is as follows. The Project Scientist requests an addition/correction in writing to the appropriate supervisor of data review/report preparation personnel, who make the requested change in a timely manner and internally verify the change. An authorized Chemist reviews and approves the addition/correction, and the Data Package Assembly Department mails or faxes the new report, which is then stored with the original data package for a minimum of ten years. In all cases, revised pages are clearly noted as such, as are additional pages added to the report.

Software Management

Triangle Labs has a formal validation program of its computer systems. Ultimately, the validation program is intended to be of a level such that all computer systems will meet the scope of any computer system audit. The validation approach is three pronged. First, new software is developed according to appropriate internal validation guidelines. Second, a validation committee has been appointed to oversee specific validation efforts of existing systems. Finally, systems are kept validated through a system of change controls. This includes the *Computer Systems Services Request* (CSSR) forms which employees use to make known to the MIS department, desired changes to software and hardware. CSSR forms include personnel sign-off for each step of the change process; and depending on the nature of the change, specify increasingly stringent required levels of authorization. Change controls also include software version control; changes to existing software are announced, uniquely labeled, documented, and old versions are archived for future reference.

The goals of the software development methodology, existing system validations, and the change control system are to ensure that the software systems perform the required functions accurately, that the users understand how to use the system, and that auditors can assure themselves of the validity of the analytical methods utilized. This in turn insures the ability to deliver accurate analyses in a timely fashion.

Section 13

DOCUMENTATION FOR QUALITY ASSURANCE

Objectives of Documentation

The objectives of documentation for quality assurance are: to provide a standardized, written program of policies, procedures and instructions; to demonstrate that adequate quality assurance and quality control procedures have been implemented; to demonstrate that accountability of the data is maintained; and to ensure traceability of analytical results.

Document Control

The laboratory maintains control over the possession and distribution of documents that directly impact the quality of a product or service. It is the responsibility of team leaders to ensure that document control files are created and maintained for all applicable documents originating in their areas. This includes documents such as the Quality Assurance Manual, Standard Operating Procedures (SOP's), Work Area Guidelines (WAGs), Quality Assurance Project Plans (QAPP's), and client instructions. It also includes standard forms, such as laboratory bench sheets, project communication forms, and corrective action reports.

A written procedure describes document control practices. Full or limited document control is applied, depending upon the purpose of the document. Those publications which document the quality assurance system at Triangle Labs, specifically the QA Manual and Standard Operating Procedures, are subject to full document control practices. Limited document control procedures are employed for other relevant documents, such as forms and flow charts . The procedure for limited document control allows for the retention of a previous version for historical information and purposes.

Every controlled document is assigned a unique identification (usually a title, file ID and creation/revision date) which must be present on each page of the document. This unique identification is entered on a master list of documents, along with a distribution list for each document to ensure that pertinent documents are made available wherever they are essential. A master set of current documents is maintained along with the master list. The status of each document, active/current or inactive/obsolete is indicated on the master list. Each document and any subsequent revisions must be reviewed and approved by authorized personnel prior to issue. Personnel authorized to review and approve a document are to have access to all necessary information on which to base their review and approval. Obsolete documents are to be retrieved from distribution points and replaced with current versions.

Standard Operating Procedures (SOP's)

Standard Operating Procedures (SOP's) are controlled documents in which instructions for standard operations performed by the laboratory are detailed. The author of an SOP should be the person most familiar with the topic being addressed. The standard format for writing SOP's is fully described in the SOP on SOPs. Each SOP is reviewed by senior level staff and authorized by management prior to distribution.

It is important that SOP's receive evaluation and input by laboratory supervisors and key technical personnel. The content of each SOPs must conform to applicable requirements of analytical methods and certification agencies, and be consistent with the Good Laboratory Practice standards. Within these constraints, the content of an SOP may be customized to meet the needs of a particular area of the laboratory. The performance of laboratory operations is subject to audit for compliance with written SOP's. If an SOP is impractical, hard to follow, or no longer meets laboratory needs, it must be modified or replaced.

The need for new or revised SOP's can be determined when a new method is implemented, when the scope of the existing method is extended or when some activities are being performed without adequate SOP's. Such a need can be identified by the analyst involved in the production or by someone from management. Also, the QA Department may identify the need and request new or revised SOP's, usually as a corrective action for deficiencies found during an internal inspection. SOP's are created to provide a clear, concise, description of the procedure with explanatory information to enable a person with the appropriate background to perform the procedure. Revisions are made to SOP's as necessary to reflect changes in procedures.

While team leaders are responsible for the operating SOPs, the administrative staff assists with the typing on an as-needed basis. Once technical approval is obtained for a new or revised SOP, the SOP is reviewed by the Quality Assurance Department for compliance with all requirements. The Quality Assurance Department also maintains a database of SOP distribution and version status, as well as maintaining the original copies of each active SOP and the historical files of each revision. The administrative staff distributes copies of the authorized SOPs to area SOP coordinators according to the distribution plan contained in the SOP database. The area SOP coordinator is responsible for discarding copies of obsolete SOPs upon receipt of revisions. Team leaders are responsible for training staff in all applicable new or revised SOPs.

Work Area Guidelines

WAGs are supplements to the SOPs and as such contain additional detail and guidance. Work Area Guidelines (WAGs) are training documents which entail step-by-step instructions for specific tasks. The WAGs are comprised almost entirely of proprietary information and are restricted to use by Triangle Labs employees. These documents cannot be distributed to clients or other non-employees.

Quality Records

Quality records must be maintained to prove that the quality assurance system is being effectively applied. At Triangle Labs, specific procedures for the identification, collection, indexing, filing, storage, maintenance, and disposition of various quality records are described in several SOP's. All quality records must be recorded in permanent (indelible) ink, legible, attributable to those personnel who wrote them, and protected so they may not be adversely affected by an unsuitable environment. They are stored and maintained in a manner that facilitates rapid retrieval for a period of at least ten years after completion. With the exception of internal audit reports, project specific quality records are available for evaluation by the client or his representative during the archive period of ten years. In fact, certain quality records, as specified by SOP or contract, are delivered to the client with the final product.

Project specific quality records are maintained to prove that adequate quality control procedures are being implemented, accountability of the project data is maintained, and traceability of analytical results is facilitated. Accountability means that reported data reflect the sample as it was received, . that sample mix-up was avoided, and the sample was properly preserved after receipt. Traceability means that reported data may be reconstructed at a later date. Through proper documentation, a laboratory is able to demonstrate or prove to clients or government agencies that the quality of the data is what the laboratory says it is. Records must contain sufficient information to permit the reconstruction of calibrations, sample preparations and sample analyses.

Quality records that are maintained at Triangle Labs include, but are not limited to, the following.

- records for sample receipt, preparation and handling
- field sample and quality control sample analysis data
- project communication tracking forms
- inspection reports for receiving, in-process and final product
- subcontractor records
- vendor qualification records
- logbooks: run logs, maintenance logs, temperature logs, balance logs, etc.
- method validation records: MDL studies, initial precision and accuracy demonstrations
- recovery data for samples, blanks and spiked samples (maintained in a database)
- system and data audit reports
- corrective action reports
- QA reports to management

Many of these quality records are discussed at length in other sections of this manual. Laboratory notebooks (or "logbooks") are utilized throughout Triangle Labs for many different purposes. All logbooks are maintained according to written procedures. New logbooks are issued by a system of signing them out in a designated logbook. Information that must be documented, both in the new logbook and the sign-out logbook, includes the assigned owner, the date issued, and the name and subject of the logbook. Logbooks must be maintained in accordance with the raw data SOP. Logbooks are kept to document all monitoring, maintenance and calibration of analytical instrumentation, and such laboratory equipment as balances, refrigerators and ovens. Software and hardware records for computers are also kept in logbooks. Logbooks specific to a piece of equipment are kept near that equipment to ensure that the work is recorded concurrently. Logbooks used for personal notes and telephone logs are distributed and tracked in the same manner as laboratory notebooks. When no longer in use, distributed notebooks are stored in the Archive Room for a minimum of ten years.

Archive

The Archive Room is locked at all times and only trained, designated staff have access. All other personnel may enter the room only in the presence of a trained Archivist and must sign and date a logbook in the Archive Room. Any materials removed from the Archive Room must be signed out by the Archivist.

All magnetic and hard copies of data, calibrations, equipment maintenance records, calculations, records of original observations, final test results and any other miscellaneous quality records directly associated with sample analyses are stored in a secured facility for a minimum ten (10) years after completion of a project. They may be stored in the Archive Room or at a secure, off-site storage facility.

Section 14

QUALITY ASSURANCE

Through a formal quality assurance system, Triangle Laboratories, Inc. is able to prove that products and services meet specific quality standards. These quality standards are defined to meet the needs and requirements of our clients, the analytical methods utilized, government agencies, and senior management of Triangle Labs.

Quality assurance is a very broad and multifaceted concept. It is composed of quality control and quality assessment. Quality control is the most important component of quality assurance. The need for quality assessment would be negligible if the laboratory always achieved perfect quality control.

> *Quality control* is a system of activities applied at each stage of the production process. Its purpose is to assure that products meet defined quality standards. This system includes the following: employee education, training, and experience; documentation (e.g., instructions, document control, records); instrument calibration and maintenance; laboratory accommodations; and inspection.
>
> *Quality assessment* is a system of activities employed to assure that quality control takes place at each stage of the production process. This system includes the following: system, data, and performance audits; reference materials; statistical evaluations; retests; and measurement bias investigation (when measurements may be operator-, instrument-, or methodology-dependent).

The success of a quality assurance system is dependent upon acknowledgment by all personnel of their responsibility for the system. The management of the laboratory is ultimately accountable for product quality, but no one person or group (e.g., the QA Department) is responsible for the greater part of quality assurance program activities. Details of the program may be found throughout this QA manual. The remainder of Section 14 will be limited to a discussion of the Quality Assurance Department, and the major activities performed and/or administered by this group.

The Quality Assurance Department

At Triangle Labs, the QA Department monitors the quality assurance system, as it is implemented throughout the laboratory, and reports the results of its observations to senior management. The Quality Assurance Manager reports directly to the President and the QA Department has no direct responsibility for production in the laboratory. The objective of this independence is to eliminate conflicts of interest in the performance of QA duties. Major activities performed and/or administered by the QA Department are summarized below. Each activity is discussed in greater detail elsewhere in the QA manual, as indicated.

- Performance of internal audits and coordination of external audits (see this section)
- Administration of a system for formal Corrective Action Reports (see this section and Section 15)
- Performance of Quality Assurance Unit (QAU) duties required for GLP-regulated studies (see this section)
- Administration of the system for document control, with emphasis on the maintenance of Standard Operating Procedures (see Section 13)
- Performance of statistical evaluations for selected quality indicators, and maintenance of quality records (e.g., control charts, summary reports) generated to document selected statistical evaluations performed throughout the laboratory (see Section 15)
- Publication of the QA Manual and other documents that describe the quality assurance system at Triangle Labs (see Section 3)

Audits and Inspections

There are several different types of audits. These may be internal, in which the laboratory reviews and examines itself, or external, in which the laboratory is audited by outside organizations, such as accrediting or regulatory agencies and clients.

Internal System Audits and Phase Inspections

A system audit is an on-site inspection and review of the quality assurance system as it is employed in the laboratory. During an audit, verification may be sought that: adequate written instructions are available for use; that analytical practices performed in the laboratory are consistent with SOP's; that adequate quality control practices are applied during production; that corrective actions are applied as necessary; that deviations from approved protocols are occurring only with proper authorization and documentation; that SOP's, quality records, analytical records, magnetic tape, etc., are properly maintained; and that personnel training records are satisfactory and current.

Internal system audits are implemented by the Quality Assurance Department to assess the functioning of one or more department(s) of the laboratory. These audits consist of real time inspections of the analytical process, comparing the daily operation to the applicable SOPs and policies. Formal inspection reports are issued detailing the extent of the inspection and any non-conformance issues noted. The production staff is required to correct all noted deficiencies and a second acceptable inspection is required for acceptance of the corrections.

Inspection reports may be routed to management at any point in the process depending on the severity of the problem. Major problems are reported to management immediately while minor ones are normally communicated in a summary report dealing with several inspections. The original of each completed inspection report, with management notification dates, is kept on file in the QA files.

Phase inspections are internal system audits that are used to verify that critical points of analysis during a pre-clinical or clinical study are being performed as specified in the applicable SOP. These inspections are performed at intervals adequate to assure the integrity of the study.

External System Audits

Representatives of clients, government agencies, and accrediting agencies frequently perform system audits of Triangle Labs. These audits are usually announced inspections, but sometimes are conducted without forewarning. QA Department personnel usually accompany such audit teams through the lab. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory and technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors typically provide an account of their findings shortly after the audit. This account is evaluated by QA personnel and reported to management, along with recommendations for actions in response to any cited deficiencies.

Data Audits

Data audits are performed by technical personnel (in Client Services or the QA Department) on a random sampling of the data reports produced at Triangle Labs. It is a goal to perform a comprehensive evaluation of a representative sampling of data reports. A data report is carefully evaluated for technical, clerical and administrative accuracy. Primary emphasis is placed on the ability of the data report to meet customer requirements. Data audits are utilized for several purposes, including: identification of opportunities for process improvement, evaluation of the efficiency of the system, detection of inadequate execution of quality control procedures, early warning of potential system deficiencies, corrective action recommendations, and reports to upper level management.

Performance Audits

A performance audit is the analysis of a fortified blank sample, for the purpose of evaluating laboratory or analyst performance. There are several examples of performance audits, which may be of internal or external origin. Performance Evaluation (PE) samples have analyte concentrations unknown to Triangle Labs, and are submitted by external organizations. PE's may be analyzed as part of multi-laboratory round robin studies, in conjunction with accreditation programs, or as blind check samples submitted by clients. Internal performance audits are fortified blanks with known analyte concentrations, the values of which may or may be known to the analyst. Examples of internal performance audits include initial precision and accuracy studies, QC check samples, laboratory control samples, and blind samples. The results of performance audits are utilized for several purposes other than the evaluation of laboratory performance, including: to fulfill accreditation requirements, to serve as analyst proficiency tests, and to facilitate laboratory improvement efforts.

Non-Conformance Reports

All instances of failure to comply with acceptance criteria are documented in a non-conformance report (NCR). Each report contains a description of the failure", details of the resulting investigation, and the determined impact on the associated sample(s). A summary of these NCR reports is reviewed by the Production Manager, the Technical Director and the Quality Assurance staff. NCRs are maintained as part of the raw data file of the project. A non-conformance issue may be caused by a particular sample independent of the analytical process or it may have been caused by a faulty analytical process with minimal adverse impact on the particular samples. The staff at Triangle strives to identify both types of situations and deal with them accordingly.

Corrective Action Reports

All major non-routine problems, deficiencies, or irregularities must be reported to management. A formal Corrective Action Report (CAR) system, administered by the QA Department, is in place at Triangle Labs. The QA Department issues CAR forms, monitors the progress of corrective actions, maintains completed documentation, and provides reports to senior management on the status of formal corrective action activities. CAR's may be originated by anyone responsible for the quality of a product. A completed form is sent to an appropriate person or group to whom responsibility for corrective action is assigned. One person is designated the Corrective Action Analyst. This person records the corrective action plans, implementations and follow-up actions completed by the responsible person(s). During the corrective action process, several measures may be taken. These include: determination of the root cause through careful analysis of processes, specifications, quality records, customer complaints, etc., using statistical process control when applicable; implementation of measures that prevent recurrence of the problem; implementation of process controls to ensure that effective corrective action is taken; application of remedial actions to products affected by the identified problem; and revision of documentation for procedures that have undergone change as a result of corrective action.

Certification and Accreditation

Triangle Labs has been granted numerous certifications and accreditations, based upon compliance with standards set forth by the granting agencies. These credentials have enabled Triangle Labs to expand and retain a substantial client base. More information about specific credentials can be found in Section 5, page 3. The nature of the quality assurance program implemented at Triangle Labs is profoundly affected by requirements of certification agencies. The administrative staff is responsible for the administration application and renewal activities associated with the various certification programs, while the QA Department is responsible for the coordination of the technical and quality issues associated with the certification programs. The QA Department is directly responsible for the coordination of:

- On-site audits by outside agencies

- Analysis of blind performance evaluation (PE) samples
- Responses to deficiencies cited in audit reports and performance evaluation results.
- Dissemination of requirements and status of certifications to relevant laboratory personnel.

GLP Regulated Studies

The Good Laboratory Practices (GLP's) are a set of regulations decreed by the United States Food and Drug Administration (FDA) and the United States Environmental Protection Agency (EPA). Compliance with these regulations is required for certain projects ("studies") completed at Triangle Labs. The GLP's define some specific responsibilities for the Quality Assurance Department. Briefly summarized, these QAU duties include the following:

- Maintenance of a copy of the master schedule sheet for all studies
- Maintenance of copies of all protocols pertaining to all studies
- Inspection of each study at adequate intervals
- Preparation of written status reports on each study with reports to management and the study director
- Determination that no deviations from approved protocols or SOP's were made without proper authorization and documentation
- Review of the final study report
- Preparation of a signed statement of the inspections performed and the dates each was reported to management for inclusion in the final study report

Section 15

QUALITY CONTROL

At Triangle Labs, quality control is achieved through the application of several procedures. Quality control activities commence before production is initiated, and are implemented at each stage of the production process. The purpose of these activities is to assure that all required standards of quality are met. Quality control activities are described in many sections of this manual. The remainder of this section will describe a subset of quality control activities that may be considered a discrete process, summarized as follows:

> Prior to the initiation of production activities, required quality standards are defined. These are derived from several sources, including: requirements of the analytical methods, needs stated by the clients, and standards established within Triangle Labs.
>
> During production, verification activities are performed to determine that defined quality standards have been met. Also, preventive measures are applied to avoid the possibility of nonconformity.
>
> When defined quality standards have not been met (nonconformities), corrective actions are applied and verified to determine that the results meet requirements.

Data Quality Objectives

Data are produced for clients at Triangle Labs. Defined quality standards for these data may be expressed as data quality objectives (DQO's). These are established prior to sample preparation and analysis. Quality assurance indicators common to all DQO's include, but are not limited to: accuracy, precision, completeness, representativeness, and comparability. Examination of the QA indicators is performed to demonstrate that the data are scientifically valid, legally defensible and that they adequately meet established DQO's. The QA indicators may be summarized as follows:

Accuracy — A quantitative measure of the relationship of reported data compared to the "true" or expected values. This measurement may be accomplished by evaluation of the recoveries of analytes spiked into samples. Specific accuracy measurement activities include surrogate spikes, matrix spikes and Quality Control Check Samples.

Precision — A quantitative measure of the reproducibility of measurements made under controlled conditions. This measurement may be accomplished by comparison of recoveries of analytes in replicate samples or injections. These analytes may be spiked or native to the duplicate samples. Specific precision measurement activities may include field replicates, lab replicates, matrix spike replicates and replicate injections.

Complete-ness A qualitative measure of the amount of valid data obtained from the analytical process compared to the amount that was expected to be obtained. Valid data must meet all data quality objectives for precision and accuracy.

Represent-ativeness A qualitative measure of the degree to which data represents the characteristics of the population from which samples were collected. This is usually dependent upon sampling techniques not controlled by the analytical laboratory. However, the issue of the representativeness of subsamples prepared within the laboratory is addressed by thorough homogenization prior to subsampling.

Comparability A qualitative measure of the confidence with which one set of data can be compared to another. Characteristics that make comparison possible include standardized report format, consistency of units (e.g., mg/L, ppm), and standardized sample preparation and analysis.

Quality Control Samples and Spikes

Analytical performance is monitored through quality control samples and spikes, such as laboratory method blanks, surrogate spikes, quality control check samples, matrix spikes, matrix spike duplicates, duplicate samples and duplicate injections. Many of these quality control measures, as applied at Triangle Labs, are summarized below.

Laboratory Method Blank A laboratory method blank consists of a sample that is processed in a manner identical to that of a regular sample, except that the matrix is replaced with distilled water for aqueous matrices, sodium sulfate for solid matrices, XAD-2 resin for MM-5 and PUF filter for PUF air sampling cartridges. The laboratory method blank sample is fortified and prepared along with the field samples, at a frequency of one laboratory method blank per batch of 20 (or less) samples of a given matrix type. The laboratory method blank serves to demonstrate a contamination free environment in the laboratory.

Surrogate Standards For certain methods, all samples, including the laboratory method blank, are spiked with a set of specific surrogate standards to monitor accuracy of the analytical determination for each particular sample. QC criteria for surrogate recoveries are method and matrix specific. Typically, laboratory QC criteria are established upon acquisition of a sufficient number of data points (20 or more) and used for evaluation of sets of data via control charts, while method specified limits are utilized for individual samples.

Quality Control Check Sample A quality control check sample consists of a blank matrix sample which is fortified not only with appropriate internal and/or surrogate standards, but also with target analytes. QC check samples are analyzed at a frequency dependent on the method. They serve as an estimation of system precision and accuracy. Results of QC check samples are monitored on control charts, with QC requirements for recoveries being established as they are for surrogate recoveries.

QC Check Sample Blank matrix spiked in the same manner as the validation series. Two (2) are analyzed each day of analysis or once per 20 samples whichever is greater. (Only 1 per day or once per 20 samples Method 1613)

Matrix Spike Sample A matrix spike (MS) sample consists of a field sample, identified by the client, that is split into two parts and processed in a manner identical to that of the rest of the field samples. However, in addition to the regular fortification with the standards (internal, surrogate and/or alternate), the chemist will add a set of the target analytes to one part of the chosen sample before the preparation. The fortification levels for the target analytes are defined by the analytical method or the client's request. At the request of the client, one such sample will be prepared for every batch of 20 samples (or less) for a given matrix. To be able to run matrix spikes, the client must provide Triangle Labs with extra sample amounts.

The analytical report for the matrix spike will contain a tabulation of the analyte concentrations as expected and as measured, along with the calculated percent recoveries based on the expected concentrations. The percent recoveries actually represent a measurement of the method accuracy for that particular sample and matrix. Accuracy is established and updated for a particular analyte and method. In the absence of observable quantitative interferences, the MS sample showing accuracies falling outside the QC limits must be reanalyzed unless the matrix spike duplicate (MSD), which was processed along with the MS, shows similar deviations as a result of a "matrix effect." This type of corrective action can only be implemented if the sample selected for the MS (and MSD) was proven to be free of the target analytes, or did not contain high concentrations that significantly exceed the MS fortification level of these analytes. "Matrix effect" is further substantiated by acceptable recoveries in a QC check sample processed along with the field samples. Matrix spike recoveries, and the possible effects on data quality when accuracies fall outside the QC limits, are discussed in the Case Narrative.

Matrix Spike Duplicate Sample The matrix spike duplicate (MSD) sample is commonly prepared (at the Client's request) in conjunction with the matrix spike (MS) sample. The analytical report will summarize the data from the MS and MSD analyses in a format allowing determination of the precision of the analyses. As for the matrix spike sample, the client must provide Triangle Labs with extra sample amounts.

Duplicate Sample A duplicate sample (DUP) consists of a set of two identical samples obtained during a single sampling session. At the client's request one such sample per batch of 20 samples (or as specified by the client) per matrix type will be analyzed, provided the client supplies Triangle Labs with the necessary samples.

The analytical report for the duplicate analyses will contain a tabulation of the results showing the precision as relative percent difference (RPD). Precision exceeding any specified target values will necessitate a non-conformance report and an evaluation of the associated data. The influence of the sampling procedure will be included in the data evaluation. The RPD is calculated as:

$$RPD = \frac{X_1 - X_2}{(X_1 + X_2)/2} \times 100$$

where: RPD = the relative percent difference

$X_i(i=1,2)$ = the analyte concentration in the original sample (1) and the duplicate sample (2)

Duplicate Injection Upon client request, a duplicate injection of a single sample extract will be performed. In the absence of observable interferences, the RPD is expected to be within ± 30% or the injections will be repeated after identification of the cause of the poor precision. Field samples analyzed during a suspected out-of-control situation will be reinjected as well.

Statistical Evaluation

Statistical evaluations can be made of selected analytical quality indicators, including spike recoveries, calibration responses, contamination levels, and method detection limits. Production units monitor levels of compliance with many criteria on a "real time" basis. Control charts are used to identify shifts in the analytical process. All identified performance shifts are investigated and causes of adverse shifts are eliminated. Causes of positive shifts are also identified and incorporated in SOPs and staff training as applicable. In-house QC criteria may be determined through historical trend analysis of data collected on QC charts. Statistical evaluations can be performed by both the QA department and production units.

QC Inspection

Quality control inspections are built into the production process. These inspections consist of peer review at each step of the process to ensure compliance with process and product specifications. Acceptance criteria are included in the production SOP's. Written documentation of the analytical process is maintained beginning with sample receipt and preparation, through instrument calibration, sample analysis, data review and report preparation. This documentation is reviewed for completeness, compliance with written procedures and consistence with client documentation.

Written records of all QC inspections are required indicating the date, inspector and results of the inspection. Detected nonconformances must be recorded during the inspection. Corrective action must be taken and documented whenever nonconformance is detected. The identity of the inspection authority responsible for releasing the product is documented in the inspection records. Until required inspections are performed on the intermediate and final product, it is not permitted to progress further along the production process, except by special, documented, client request.

In-process Inspection and Testing

Each department is responsible for a segment of the production process and for all in-process inspection and testing that takes place within the department. In-process inspection is accomplished through 100% screening for all areas. Each client sample that goes through the analytical process is unique and can be considered a separate lot.

Final Inspection and Testing

The last stage of the production process is the preparation of a final data report. This requires a thorough review of all records generated for a client sample set since its receipt, including inspection records and any client documentation that may have originated before sample receipt. A chemist performs this function during the preparation of the data package. This inspection serves as both an in-process and final inspection of the product. In addition, a second chemist performs another final inspection of the data package and quality records. As in any other part of the process, any nonconformances found during these inspections must be documented and corrected before the data package is released. Approval of the data package for release to the client is indicated by the signatures of the chemists on the case narrative.

Nonconformity

Each field sample that is incorporated into the analytical process is unique. Laboratory procedures are designed to introduce as much standardization as possible. Whenever conformance to standards is uncertain, the product is reviewed to determine the nature and cause of nonconformance. If it is judged to be nonconforming due to the unique nature of a sample, there may be little recourse other than to inform the client and discuss the options that are available.

Each case of failure to comply with written acceptance criteria must be recorded in a non-conformance report (NCR). The failure must be recorded by the person who detected or observed it. All investigative efforts are recorded on the NCR with an evaluation of the impact the non-conformance had on the associated samples. Impact on the analytical process is also noted. If needed, recommendations for corrective action are made. A copy of the NCR is kept with the project data. Rework and reanalysis is subject to the same inspection procedures as the initial work. Nonconformity, its review, and its disposition must be documented in the quality records as prescribed by the written procedures.

Corrective and Preventive Action

Appropriate actions must be taken to prevent or correct nonconformities in products and problems in analytical systems. When actions result in permanent procedural changes, pertinent documentation (e.g., SOP's) must also be modified to reflect these changes. Cost-effective preventive measures are applied whenever possible. In specific cases, the cost of applying preventive measures would exceed the cost of applying routine corrective actions. Because every client sample possesses unique and unknown properties, some predisposition to unpredictable, unpreventable nonconformities exists.

Corrective Action

Specific corrective actions are of two types: routine corrective actions applied to solve minor or commonplace problems, and formal corrective actions taken to eliminate major or non-routine problems.

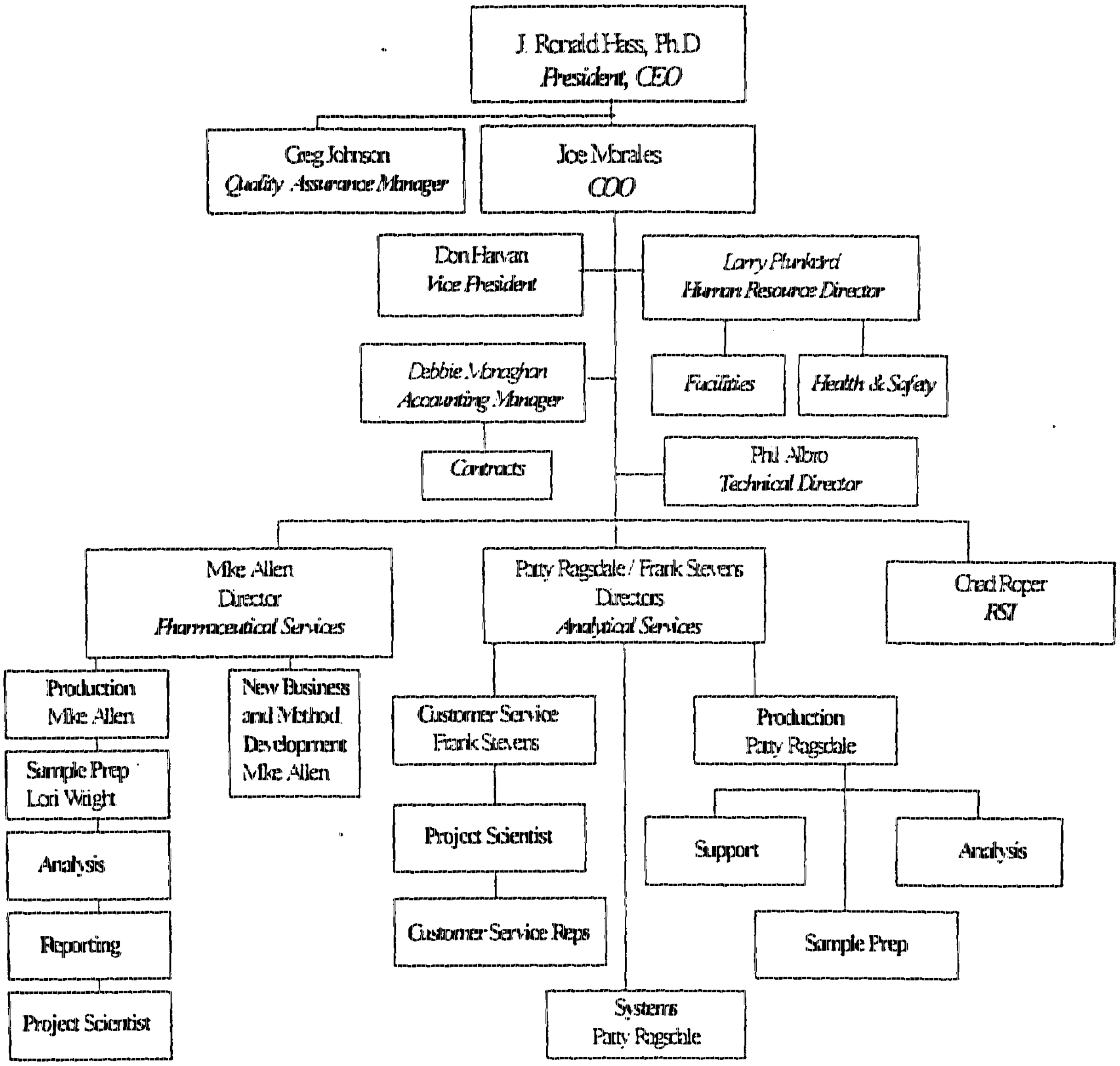
Routine corrective actions are usually made by the chemists, technicians or instrument operators who detect minor problems or product nonconformances. These actions are taken in response to observed non-conformance issues are recorded on the associated NCRs.

There are three procedures for conducting formal corrective actions. The first is corrective action in response to a system audit report from the Quality Assurance Unit. This procedure is more thoroughly described in Section 14. The second procedure is the formal Corrective Action Report, which may be initiated by anyone who detects a significant quality problem. This procedure is also administered by the Quality Assurance Unit. Further information about it can be found in Section 14. The third practice is described in a written procedure on "Problem Sample Communication." It is initiated in response to client complaints about specific projects.

Preventive Action

Preventive actions are implemented as part of standard operating procedures, process improvement efforts and corrective actions. When circumstances inherent to a procedure are known to have a high potential for error, the SOP must define measures to prevent the error from occurring. Preventive actions are an integral part of corrective actions, because resultant changes in procedures often prevent recurrence of problems.

Triangle Laboratories, Inc.



TRIANGLE LABORATORIES, INC.
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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290

TLI SOP No. DSP105	Version: 15	Effective Date:	July 31, 1998
Author:	Don Harvan	Date Written:	May 4, 1998
Authorization:	Phil Albro (Management)	Date Authorized:	5/27/98

I. **SCOPE AND APPLICATION:** This method provides procedures for the extraction of polychlorinated dibenzo-p-dioxin (tetra- through octachlorinated homologues; PCDDs) and polychlorinated dibenzofuran (tetra- through octachlorinated homologues; PCDFs) from non-tissue solids, including but not limited to soil, sediment, pulp, sludge, paper and/or cardboard, according to SW-846 Method 8290. Only the extraction procedure is contained in this SOP.

II. **SAFETY CONSIDERATIONS:** Samples may contain harmful substances. Wear labcoat, appropriate eyewear and gloves. Toluene and ethanol are flammable; avoid flames and sparks. The procedure has been validated for either heptane or hexane, since heptane is much less of a health hazard, it should be used instead of hexane whenever possible, but hexane can be used if heptane is unavailable. Do not breathe the vapors, and avoid contact with skin or eyes, as these solvents may cause irritation, nausea, and dizziness. For additional safety information, see the TLI Safety and Health Manual and the appropriate MSDS.

NOTES: **Slight procedural differences exist for specified sample types in this SOP. Be careful not to overlook these differences.**

III. **REAGENTS:**

A. Heptane- pesticide grade (< 1 ppm residue)

B. Toluene- pesticide grade (< 1 ppm residue)

C. Ethanol- OmnySolve grade

D. Ethanol/toluene (68/32 v/v) - Take 68 parts (by volume) of Ethanol and mix well with 32 parts (by volume) of Toluene.

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
TLI SOP No. DSP105 Version: 15	Date Written: May 4, 1998

IV. GLASSWARE PREPARATION

A. All glassware used in this procedure must have been prepared according to glassware washing SOP.

B. The following glassware is needed for each sample:

1. beaker
2. 500 mL or 250 mL flat bottom flask
3. forceps
4. spatula
5. thimble holder - pre-Soxhlet extracted and ready to use
6. Soxhlet extractor - pre-Soxhlet extracted and ready to use

C. If wet sample ≥ 15 g, Soxhlet Dean Stark (SDS) extractor must be used in addition to the Soxhlet extractor. Be sure the SDS extractor has been pre-Soxhlet extracted.

D. The flask, thimble holder and Soxhlet or SDS extractor may be 250 or 500 mL, but all pieces must be the same size.

E. If SDS is required, assemble the SDS extractor with stopcock.

F. Rinse all glassware with n-heptane.

G. Label each beaker and flask by placing a color coded sample label on top of a strip of colored lab tape. The colored lab tape is necessary because the adhesive on the color coded labels is difficult to remove from the glass and will cause contamination problems.

H. All flasks require two labels - one on the side of the flask and one on the neck.

V. EXTRACTION PROCEDURE:

A. Plan the extraction batch. An extraction batch can contain up to twenty (20) samples and must include a method blank, a spike pair, and at least one(1) Lab

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
TLI SOP No. DSP105 Version: 15	Date Written: May 4, 1998

Control Spike (LCS). Complete the QC batch form, including all samples and QC samples in the extraction batch.

B. Observe and document the physical appearance and consistency of each sample in the wet lab observation log in MILES.

C. Using the percent moisture analysis, determine the amount of sample necessary for a 10 g dry weight sample.

Note: Mix the sample well before taking an aliquot for extraction.

D. For each sample, weigh the amount of sample determined in step VI.C. above. Record the sample weight on the Sample Preparation Management and Tracking Form (SPMFT).

E. Make sure homogeneity is maintained--exclude anything that does not constitute sample's natural matrix.

F. Clean balance by swiping with heptane.

G. Zero balance. Weigh sample and its container.

H. Record the gross weight on the STMF (Sample Tracking and Management Form).

I. Tare beaker in which sample is to be transferred.

J. Record the sample weight on the STMF (Sample Tracking and Management Form). Also indicate with the letter"Y" or "N" if all the sample was used.

(Y)=There is enough sample left to perform re-extraction
(N)=There is NOT enough sample left to perform re-extraction

K. Reweigh sample and its container weight. Record the post-gross weight.

L. Prepare the blank using pre-extracted G-8 filters for pulps and all dried and ground samples. Prepare the blank using pre-extracted sand for all normal ash/sediment/soil/sludge samples. If any laboratory control samples (LCS and/or

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
TLI SOP No. DSP105 Version: 15	Date Written: May 4, 1998

LCSD) are included in the batch, use the same matrix as Blank. Weigh out 10 grams (± 0.4) for each blank and/or OPR prepared.

M. Check the volume of each standard solution relative to the last volume mark. The bottom of the meniscus must be at the mark.

N. Spike all samples with 20 µL USF-I (0.1µg/mL). Spike any Lab Control Spikes,(LCS/LCSD), Matrix Spikes (MS/MSD) with 40 µL USF-MX (0.01 µg/mL). Record the volume, lot number, concentration and expiration date of each standard on the SPMFT and initial and date the entry. At the end of the spiking process, mark the bottom of the meniscus with a fine point permanent marking pen. Return the standard vials to the storage drawer.

Note: Dioxin standards must be stored at room temperature in amber, glass vials, with Teflon lined septa caps. Spiking instructions can be found in See SOP on Concentration of Extracts Using Rotary Evaporator.

O. After spiking place a glass wool plug on top of the sample in each thimble. Use pre-extracted glasswool.

P. Prepare the Soxhlet apparatus by:

1. Use a 500 mL setup, place 400 mL toluene* in the flatbottom flask. Add 5-6 Teflon boiling chips in each flask.
2. Place the label containing information for the concentration process on the boiling flask on top of the colored lab tape. Write the solvent used on the flask.
3. Place the pre-Soxhleted thimble holder on top of the flask.

Q. ***NOTE:** GP pulp sludge samples require 68:32 ethanol/toluene instead of toluene. GP pulp samples require ethanol instead of toluene. The volume remains 400 mL for 500 mL setup.

R. Place the thimble with the spike sample into the thimble holder. Be sure to seat the thimble at the bottom of the holder.

S. **NOTE:** While the Soxhlet apparatus is sitting on the counter, keep the top capped with aluminum foil to avoid contamination.

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
TLI SOP No. DSP105 Version: 15	Date Written: May 4, 1998

T. Place the Soxhlet on top of the thimble holder. If Soxhlet Dean Stark (SDS) is being used, be sure the stopcock is closed.

U. Place the Soxhlet apparatus on the heating mantle and connect the Soxhlet apparatus to the condenser at the ground glass joint. Cap the top of the condenser with aluminum foil.

V. Wrap the thimble holder and Soxhlet extractor with foil. Do not cover the condenser.

W. **NOTE:** If using ethanol as the sole solvent, do not wrap any of the apparatus with aluminum foil.

X. Place a piece of colored tape on the condenser to indicate it is being used for a sample and needs to be pre-Soxhlet extracted after the sample extraction is completed.

Y. If the samples have been designated **high level** write **2X** on the tape. If samples have designated **Isolation** write **3X** on the tape.

Z. Turn on the heat. If using a hot plate, use the heat setting #5. If using a six (6) place mantle, use the HIGH setting.

AA. Check the units after one (1) hour. Each unit should be cycling at a rate of five (5) times per hour, have no leaks and have sufficient solvent. Open the aluminum foil on the thimble holder to check the cycling action and close the wrapping. Make adjustments as necessary.

BB. If adjustments were required, check the units again in one (1) hour. Otherwise check again in six (6) hours.

CC. If the weighed sample is >30g, drain the water from the SDS halfway through the extraction (i.e., approximately 8 hours after turning on the heaters).

DD. Extract the sample for 16 hours.

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
TLI SOP No. DSP105 Version: 15	Date Written: May 4, 1998

TRIANGLE LABORATORIES, INC. PAGE 1 OF 1

Dioxin Sample Preparation Tracking & Management Form

Project: 91500 Client: Triangle Laboratories, Inc. (TLI01)

Solvent(s)/Acid(s): ______ / ______ / ______ Method: 8290E.Tetra-octa Matrix: ______

Lot Numbers: ______ / ______ / ______ Extraction Date: __/__/__

IS Spike: 20 ul conc: 0.1000 ng/ul
SS Spike: ____ ul conc: ____ ng/ul
AE Spike: 0 ul conc: 0.0000 ng/ul
LCS Spike: 0 ul conc: 0.0000 ng/ul

______	______	______	______	Chemist
______	______	______	______	Spike #
______	______	______	______	* ID
__/__/__	__/__/__	__/__/__	__/__/__	* Expir
__/__/__	__/__/__	__/__/__	__/__/__	* Date
__:__	__:__	__:__	__:__	* Time
____ng/ul	____ng/ul	____ng/ul	____ng/ul	* Conc.
____ ul	____ ul	____ ul	____ ul	* Vol.

No.crd	TLI SAMPLE ID / CLIENT SAMPLE ID	GROSS WEIGHT Before	GROSS WEIGHT After	SAMPLE SIZE g / ml					
000	TLI Blank / TLI Method Blank								Any Left? yes/no
									Any Left? yes/no
									Any Left? yes/no
									Any Left? yes/no
									Any Left? yes/no
									Any Left? yes/no
									Any Left? yes/no
									Any Left? yes/no
									Any Left? yes/no
									Any Left? yes/no

Gross weight of sample container + sample before/after aliquot removal.

Comments: ______________________________

____

____

______________________________ Initials: ______ Date: __/__/__

Initials of both SPIKER AND OBSERVER must be entered. XXXX = Gross weight not provided for OTHER Samples.

REV 10/27/97 (PTSMF 7)

ATTACHMENT 1

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
TLI SOP No. DSP105 Version: 15	Date Written: May 4, 1998

Composition of Fortification Standards

USF-I

Analyte	Concentration (µg/mL)
13C, 2,3,7,8-TCDF	0.1
13C, 2,3,7,8-TCDD	0.1
13C, 1,2,3,7,8-PeCDF	0.1
13C, 1,2,3,7,8-PeCDD	0.1
13C, 1,2,3,6,7,8-HxCDF	0.1
13C, 1,2,3,6,7,8-HxCDD	0.1
13C, 1,2,3,4,6,7,8-HpCDF	0.1
13C, 1,2,3,4,6,7,8-HpCDD	0.1
13C-OCDD	0.2

USF-MX

	Concentration (µg/mL)
3-MonoCDF	0.01
3-MonoCDD	0.01
2,3-DiCDF	0.01
2,3-DiCDD	0.01
2,3,8-TriCDF	0.01
1,2,4-TriCDD	0.01
2,3,7,8-TCDF	0.01
2,3,7,8-TCDD	0.01
1,2,3,7,8-PeCDF	0.05
2,3,4,7,8-PeCDF	0.05
1,2,3,7,8-PeCDD	0.05
1,2,3,4,7,8-HxCDF	0.05
1,2,3,6,7,8-HxCDF	0.05
1,2,3,7,8,9-HxCDF	0.05
2,3,4,6,7,8-HxCDF	0.05
1,2,3,4,7,8-HxCDD	0.05
1,2,3,6,7,8-HxCDD	0.05
1,2,3,7,8,9-HxCDD	0.05
1,2,3,4,6,7,8-HpCDF	0.05
1,2,3,4,7,8,9-HpCDF	0.05
1,2,3,4,6,7,8-HpCDD	0.05
OCDF	0.1
OCDD	0.1

USF-C

Analyte	Concentration (µg/mL)
37Cl- 2,3,7,8-TCDD	0.01

ATTACHMENT 2

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TOTAL PERCENT SOLID DETERMINATION			
TLI SOP No. DSP210	Version: 6	Effective Date:	3/26/99
Author:	Margaret Kabala	Date Written:	March 8, 1999
Authorization:	Phil Albro Management	Date Authorized:	3/19/99

I. **SCOPE AND APPLICATION:** The purpose of this SOP is to describe the procedure on how to determine the percent solid in water samples.

II. **PROCEDURE:**

A. Bake an empty (w/o cap) 15 mL centrifuge tube in the oven for at least one hour.

B. Label tube with project number, TLI number and sample number using a red or black marker. Label cap using a color coded sample label.

C. Weigh the empty tube with cap to the nearest $\pm$ 0.01 gm.

D. Shake the jar with the sample for 2 minutes.

E. Immediately draw off 10 mL aliquot from the jar and transfer into a pre-weighed 15 mL centrifuge tube.

F. Weigh the tube with the sample and cap to the nearest 0.01 g.

G. Centrifuge the sample at 1500 rpm for 15 minutes.

H. Decant the supernatant layer. Place the tubes in oven without the caps.

I. Bake the sample in the oven fisher No. 506 G at 110±5°C for at least 12 hours.

J. Remove sample from the oven and let cool in a dessicator.

K. Weigh the dried sample and the tube with the cap.

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PERCENT SOLID DETERMINATION	
TLI SOP No. DSP210 Version: 6	Date Written: March 8, 1999

L. Return the sample in the oven for another 3 hours.

M. Repeat steps H to L until the weight is constant.

N. Record all the weights in MILES.

O. A computer calculation of percent moisture can be obtained by performing the following steps:

-log onto MILES and go to wet lab

-go to Result Entry- type in project number and extension (if any)

-check samples that require % solids; press F10

-type in weights and make sure to verify all entries

-press enter or page down to save all entries

-to print results, go to results report

-select "M" for moistures

-select 2 to print using the wet lab laser printer

-check samples then press F10

-get printout from laser printer

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PREPARATION AND EXTRACTION OF FISH SAMPLES FOR PCDD/PCDF				
TLI SOP No.	DSP130	Version: 11	Effective Date:	4/30/99
Author:	Heather Brown		Date Written:	March 11, 1999
Authorization:	Phil Albro (Management)		Date Authorized:	4/19/99

I. SCOPE AND APPLICATION: The purpose of this SOP is to describe the procedure for filleting fish, subsampling and extraction of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans according to Methods 1613B and 8290.

II. SAFETY CONSIDERATIONS: Wear lab coat, goggles, gloves, and mask. Take care not to injure yourself with knife or grinder. Avoid breathing solvent. This can cause dizziness, nausea, and in extreme cases, death. For additional safety information, see the TLI Safety and Health manual and the appropriate MSDS.

III. PROCEDURE FOR FILETING:

A. Remove fish scales using an electric fish scaler and skin as per client requirement(s).

B. Place the fish on clean filleting board with acetone and hexane-rinsed aluminum foil (dull side facing the sample).

C. Using a filleting knife (which has been washed with soap and water then rinsed with acetone and heptane), make a cut on the left side of the dorsal fin just above the rib cage and along the spine.

D. Remove the fillet without the rib bones.

E. On the other side of the fish, repeat steps B to D.

F. Put each fillet on the dull side of a decontaminated aluminum foil.

G. Weigh aluminum foil, each fillet indicating the weight of the left and right sides of the fish.

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PREPARATION AND EXTRACTION OF FISH SAMPLES FOR PCDD/PCDF	
TLI SOP No. DSP130 Version: 11	Date Written: March 11, 1999

H. Record in the sample weights log book the weight of each fillet from each individual fish.

I. Get the total weight of all fillets included in one whole composite sample.

J. Grind the filleted sample using Hobart grinder or blender.

K. Weigh 25 g of the sample for extraction, if lipids are to be done. Weigh 20 g of sample for extraction if no lipids are to be done.

L. Wrap separately the remaining ground and ungrounded samples in clean aluminum foil, then seal them in clean, labeled plastic bags.

M. Keep the samples in a - 12°C freezer.

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PREPARATION AND EXTRACTION OF FISH SAMPLES FOR PCDD/PCDF	
TLI SOP No. DSP130 Version: 11	Date Written: March 11, 1999

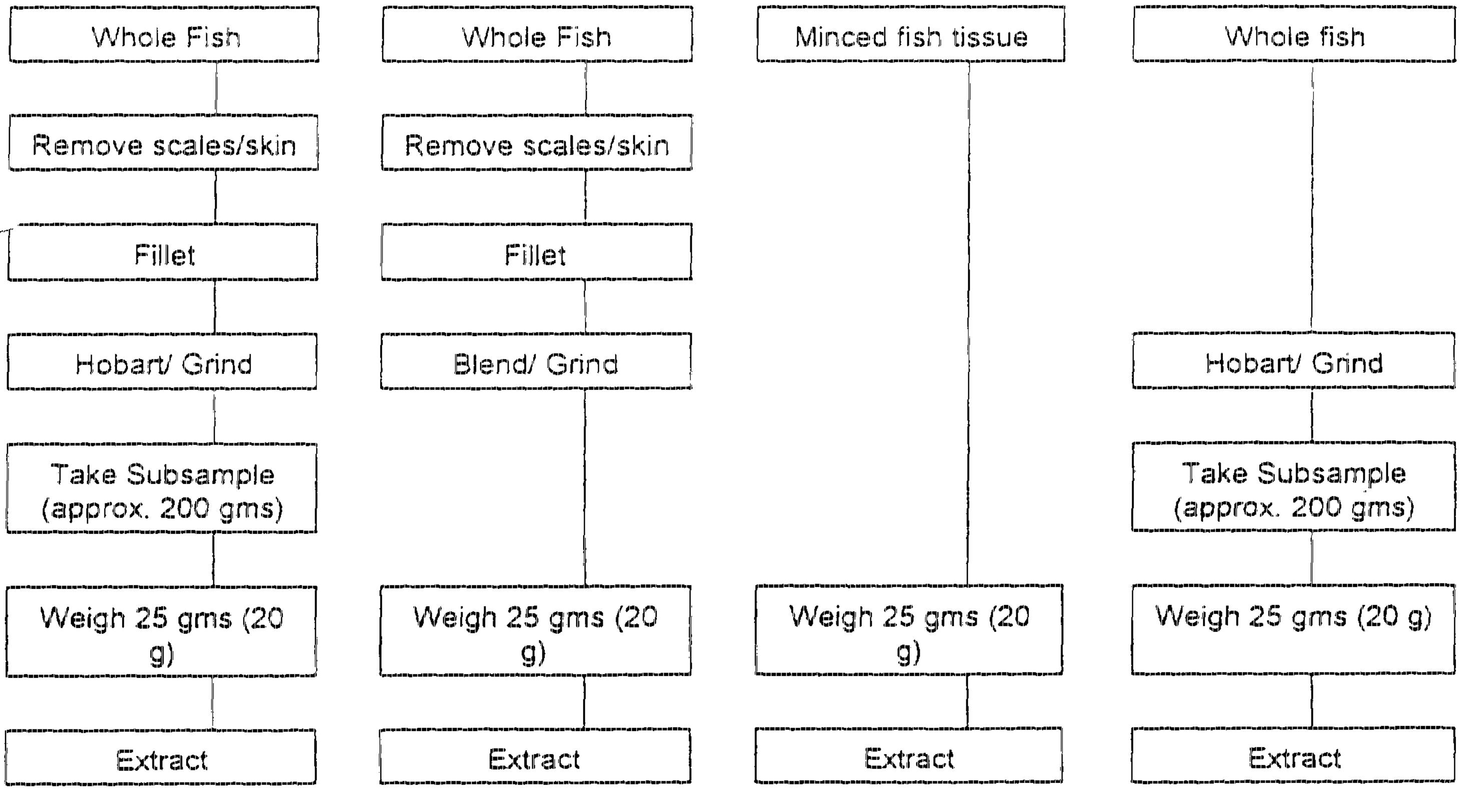
PREPARATION OF FISH SAMPLES FOR EXTRACTION

A. Fillet from large fish or fish large fillet composite or small fish (200 gms or more)

B. Fillet from small fish (100 - 200 gms)

C. Minced fish tissue (approx. 100 gms)

D. Whole (large including composites)



PREPARATION AND EXTRACTION OF FISH SAMPLES FOR PCDD/PCDF	
TLI SOP No. DSP130 Version: 11	Date Written: March 11, 1999

I. PROCEDURE FOR SUBSAMPLING:

A. Spread the blended/ground fish tissue composite on clean aluminum foil placed on top of a flat bench.

B. Quarter the sample using a clean knife or spatula.

C. Take two diagonal quarters and mix them thoroughly to produce a subsample. Care must be taken not to exclude skin in the quartering process.

D. Repeat steps B and C until a subsample of 200 gm is left.

E. Tissumize the subsample until a slurry-like composition is attained.

F. Weigh the desired amount of the tissumized subsample for extraction.

G. Wrap the remaining tissumized subsample in clean, labeled aluminum foil.

H. Store the subsample together with the original composite of the same sample in a sealed plastic bag in a -12°C in a freezer.

I. Label the plastic bag with Project #, TLI ID, Lab # and Client ID.

II. PROCEDURE FOR EXTRACTION:

Transfer 20 g (if no lipid determination) or 25g (if lipid determination) of the Tissumized fish to a precleaned Soxhlet thimble. Add 75 g of sodium sulfate, and mix well in the thimble. For the blank, use 75 grams of sodium sulfate. For 1613B, add 0.5 mL of corn oil to the blank and OPR.

A. Spike the fish/sodium sulfate mixture with the appropriate amount of internal standard specified on the Sample Management Tracking Form. Note that the sample size for PCDD/PCDF analysis is 20 g whether lipid is to be determined or not.

B. Soxhlet extract for 16 hours using Methylene Chloride/Heptane ,3:1 (v/v).

C. If lipids are NOT to be determined, skip step D. and jump to step E.

D. If lipids are to be determined, rotovap to approximately 10 mL, and then transfer the sample, using methylene chloride rinses, to a 50-mL graduated centrifuge

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PREPARATION AND EXTRACTION OF FISH SAMPLES FOR PCDD/PCDF	
TLI SOP No. DSP130 Version: 11	Date Written: March 11, 1999

tube. Save sample flasks to use later. Fill the tube with methylene chloride to exactly 25 mL. Spike the sample with the appropriate spike surrogate/ alternate standards for a 25 g sample, mix well using a vortex, then pipet 5 mL into a pre-weighed scintillation vial for lipid determination. Transfer the remaining 20mL back to the original flask. Rinse the 50 mL tube twice with methylene chloride and transfer the rinsings to the flask.

E. For 8290, add 0.5 mL of tridecane to the flasks for all samples (not the scintillation vials). Tridecane is not needed for 1613B since all samples contain protective lipids. Rotary evaporate to dryness. Solvent exchange twice with 20 mL each time of n-heptane or isooctane. Relinquish to cleanup. Follow SOPs DSP 280 and DSP260.

F. Evaporate the 5 mL of the sample solution from D. above to constant weight using nitrogen.

G. Reweigh the dried sample in the scintillation vial.

H. Record all the weights in the sample weight logbook and % lipid form.

I. Calculation:

$$\%\ \text{lipids} = \frac{(\text{Wt. of dried sample + vial in g}) - (\text{Wt. of vial in g})}{\text{Weight of Sample}/5} \times 100$$

NOTE: Initially we will need to stop after concentration just prior to the clean-up stage. Observe extract to determine if clean-up column will be overloaded with lipids.

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EXTRACTION OF PCDD/PCDF FROM FISH TISSUE - METHODS 1613 & 8290

TLI SOP No. DSP230	Version: 5	Effective Date:	June 8 1998
Author:	Anzor Gatchetchiladze	Date Written:	January 27, 1998
Authorization:	Phil Albro (Management)	Date Authorized:	5/15/98

I. **SCOPE AND APPLICATION:** The purpose of this SOP is to describe the use of the Tissumizer[R] to prepare fish for analysis of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans. It follows the procedure designated by the State of Washington. The lipid analytical procedure designated by that state is also described.

II. **SAFETY CONSIDERATIONS:** Wear labcoat, appropriate eyewear, and gloves. Avoid breathing methylene chloride vapor, as it can cause dizziness, nausea, irritation, and, in extreme cases, death. For additional safety information, see the TLI Safety and Health Manual and the appropriate MSDS.

III. **PROCEDURE:**

A. Prepare the fish as described in the SOP on Filleting Fish and Subsampling Fish.

B. Weigh 25 grams of the homogenized fish sample, and place it into a methylene chloride thimble soxhlet extracted with methylene chloride for Method 8290 or Toluene for Method 1613. Add 75 grams of methylene chloride presoxed sodium sulfate, and mix it well with the fish sample in the thimble. In case the fish sample is very wet, place it in a clean beaker (instead of thimble), and add as much sodium sulfate until a mixture with sandy consistency is achieved. Then transfer the mixture into the thimble. Use 75 grams sodium sulfate for the blank and OPR.

C. Spike all samples with the appropriate amount of internal standard (specified on the Sample Preparation Tracking and Management Form). Record the volume, lot number concentration and expiration date of each standard on the SPTMF and initial and date.

For 8290: Spike blank and all samples with 25µL of USX-I (0.1µg/ml), LCS, LCSD, MS, MSD with 50µL of USX-MX (0.01µg/ml).

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EXTRACTION OF PCDD/PCDF FROM FISH TISSUE - METHODS 1613 & 8290	
TLI SOP No. DSP230 Version: 5	Date Written: January 27, 1998

For 1613: Spike blank and all the samples with 25µL USX-AIS (0.1µg/ml), spike the OPR with 25µL USX-MX (0.01µg/ml). Spike any MS/MSD with 50µL USX-MX (0.01µg/ml). If only a 20 gram sample is used for extraction, spike samples with 20µL of USX-AIS (0.1µg/ml), 20µL of USX-MX (0.01µg/ml) for the OPR and 40µL of USX-MX (0.01µg/ml) for the (MS/MSD).

D. Soxhlet extract the fish sample for 16 hours using 400 mL methylene chloride for Westvaco and 370 mL toluene for any other client. Use Dean Stark soxhlet extractors (SDS) if toluene is used as the extracting solvent.

E. After extraction, discard water from the SDS traps. Rotovap extract to 10 mL. Then transfer the extract into 50 mL graduated centrifuge tube using extraction solvent rinses. Fill the tube to exactly 25 mL. (**NOTE:** Save sample flasks and use them during the Big Fish column clean-up).

F. Spike the extract with 25µL of USX-C (0.01µg/mL) as indicated in the SPTMF. Vortex the spiked extract. Immediately pipet 5 mL of the extract, and add it into a preweighed scintillation vial for lipid determination. Add 500 µL n-Tridecane to the blank and OPR (On-going Performance and Recovery) samples only.

G. Transfer the remaining 20 mL extract back into the original 500 mL flat-bottom flask, then add tridecane. Samples should be concentrated to dryness then solvent exchanged twice with iso-octane. Samples are then ready for clean-up.

H. Follow SOP on Column Chromatography for Dioxin Extracts.for cleanup.

1. LIPID DETERMINATION:

   a) Evaporate the 5 mL sample (step F above) in the scintillation vial to dryness. After drying the solvent in the blank and OPR samples, allow other samples to dry for another 10-15 minutes.

   b) Weigh the vial with the lipid. Dry again using Nitrogen for another 10-15 minutes. Weigh the vial with the lipid a second time to check if constant weight is achieved.

   c) Record the weights in the sample logbook and the % lipid form.

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EXTRACTION OF PCDD/PCDF FROM FISH TISSUE - METHODS 1613 & 8290	
TLI SOP No. DSP230 Version: 5	Date Written: January 27, 1998

d) Calculation:

$$\% \text{ lipids} = \frac{(\text{Wt. of dried sample + vial in g}) - (\text{Wt. of vial in g})}{\text{Weight of Sample/5}} \times 100$$

NOTE: Initially we will need to stop after concentration just prior to the clean-up stage. Observe extract to determine if clean-up column will be overloaded with lipids.

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FILLETING FISH FOR DIOXIN EXTRACTION

TLI SOP No. DSP195	Version: 5	Effective Date:	4/8/99
Author:	Louise Bailey	Date Written:	March 11, 1999
Authorization:	[signature] Management	Date Authorized:	3/26/99

I. **SCOPE AND APPLICATION:** The purpose of this SOP is to describe the procedure for filleting fish for extraction of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans.

II. **SAFETY CONSIDERATIONS:** Wear lab coat, goggles, gloves, and mask. Take care not to injure yourself with knife or grinder. Avoid breathing solvent. This can cause dizziness. nausea, and in extreme cases, death. For additional safety information, see the TLI Safety and Health manual and the appropriate MSDS.

III. **PROCEDURE:**

A. Remove fish scales using an electric fish scaler and skin as per client requirement(s).

B. Place the fish on clean filleting board with acetone and heptane-rinsed aluminum foil (dull side facing the sample).

C. Using a filleting knife (which has been washed with soap and water then rinsed with acetone and heptane), make a cut on the left side of the dorsal fin just above the rib cage and along the spine.

D. Remove the fillet without the rib bones.

E. On the other side of the fish, repeat steps B to D.

F. Put each fillet on the dull side of a decontaminated aluminum foil.

G. Weigh aluminum foil, each fillet indicating the weight of the left and right sides of the fish.

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FILLETING FISH FOR DIOXIN EXTRACTION	
TLI SOP No. DSP195 Version: 5	Date Written: March 11, 1999

H. Record in the sample weights log book the weight of each fillet from each individual fish.

I. Get the total weight of all fillets included in one whole composite sample.

J. Grind the filleted sample using Hobart grinder or blender.

K. Weigh 25 g of the sample for extraction, if lipids are to be done. Weigh 20 g of sample for extraction if no lipids are to be done.

L. Wrap separately the remaining ground and ungrounded samples in clean aluminum foil, then seal them in clean, labeled plastic bags.

M. Keep the samples in a - 12°C freezer.

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FILLETING FISH FOR DIOXIN EXTRACTION	
TLI SOP No. DSP195 Version: 5	Date Written: March 11, 1999

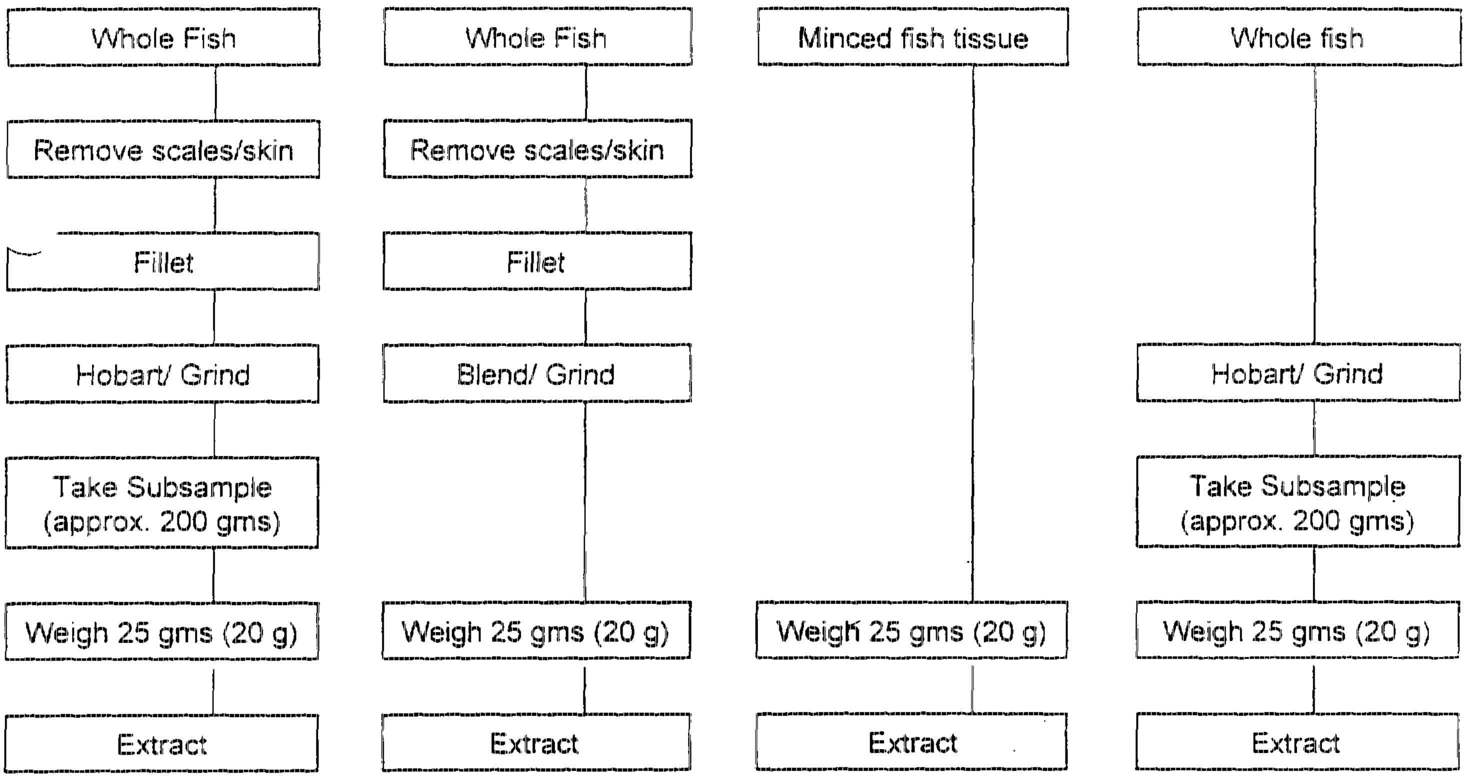
PREPARATION OF FISH SAMPLES FOR EXTRACTION

A. Fillet from large fish or fish large fillet composite or small fish (200 gms or more)

B. Fillet from small fish (100 - 200 gms)

C. Minced fish tissue approx. 100 gms)

D. Whole (large including composites)



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SUBSAMPLING FISH SAMPLE FOR EXTRACTION			
TLI SOP No. DSP192 Version: 3		Effective Date:	July 9, 1997
Author:	Belen Rueda	Date Written:	June 3, 1997
Technical Approval:	Phil Albro (Technical Director)	Date Approved:	6/25/97
Authorization:	[signature] (Production Manager)	Date Authorized:	6/30/97

I. **SCOPE AND APPLICATION:** The purpose of this SOP is to describe the procedure for subsampling a fish sample for extraction and clean-up of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans for analysis by GC/MS.

II. **PROCEDURE:**

A. Spread the blended/grounded fish tissue composite on clean aluminum foil placed on top of a flat bench.

B. Quarter the sample using a clean knife or spatula.

C. Take two diagonal quarters and mix them thoroughly to produce a subsample. Care must be taken not to exclude skin in the quartering process.

D. Repeat steps B and C until a subsample of 200 gm is left.

E. Weigh the desired amount of the subsample for extraction.

F. Wrap the remaining subsample in clean, labeled aluminum foil.

G. Store the subsample together with the original composite of the same sample in a sealed plastic bag in a - 20°C freezer.

H. Label the plastic bag with Project #, TLI ID, Lab # and Client ID.

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EXTRACTION OF BIOLOGICAL MATERIAL USING WARING BLENDER	
TLI SOP No. DSP293 Version: 1	Date Written: 18 November 1999

Author: Annette Suing | Effective Date: 12/7/99

Authorization: Phil Albro (Management) | Date Authorized: 11/30/99

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I. **PURPOSE:** The purpose of this SOP is to describe the procedure to be followed when samples are extracted with a Waring blender for PCDD/PCDF or PCB analysis.

II. **SCOPE:** This procedure is applicable for the extraction of total lipids and lipid soluble analytes from soft, deboned tissues or processed foods when the water content is 80-90% of the sample weight. Total lipid may be determined easily from the extract, if desired.

III. **SAFETY CONSIDERATIONS:** Samples may contain harmful substances. Wear labcoat, appropriate eyewear, and gloves. Do not breathe the solvent vapors, and avoid contact with skin and eyes. The solvents may cause dizziness, irritation, nausea, and in extreme cases, death. Solvents used in this procedure are not highly flammable, but if they should be ignited it is essential to avoid breathing the gas emitted (could contain phosgene.) For additional safety and health information, see the TLI Safety and Health Manual and appropriate MSDS forms.

IV. **PROCEDURE:**

A. INITIAL EXTRACTION:

1. Weigh out 20 ± 1 grams of sample. Put weighed sample in a stainless steel blender canister capable of holding at least 500 mL of solvent. Use 0.1 mL of corn oil as the blank, OPR or LCS, if the samples are considered low-fat (less than 3% lipid), or 0.5 mL of corn oil if the samples are considered high fat (greater than 3% lipid) or the lipid content is unknown.

2. Spike with internal standard mixture as specified in the project folder.

3. Add 400 mL of methylene chloride: methanol, 2:1 (v/v). This must be measured accurately.

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EXTRACTION OF BIOLOGICAL MATERIAL USING WARING BLENDER	
TLI SOP No. DSP293 Version: 1	Date Revised: 18 November 1999

NOTE 1: For other sample sizes, use 20 mL of solvent mixture per gram of tissue.

4. Cap the canister tightly with a solvent-resistant cover (not the plastic cover supplied with the blenders.) A foil-covered plastic cover is acceptable if the solvent can not come in contact with the plastic.

NOTE 2: It is preferable to have the blender motor connected to a Variac so that the voltage can be brought up slowly to avoid violent splashing.

5. Blend for 30 seconds. After turning off the power, wait 30 seconds for settling.

6. Pour the extract onto a pre-cleaned G8 glass fiber filter in a porcelain vacuum funnel coupled to a clean filtering flask large enough to hold at least 600 mL of solvent without risk of overflow into the sidearm.

NOTE 3: A Type A filter can be used if G8 filters are not available.

7. Vacuum filter until dripping stops.

NOTE 4: Turn off the vacuum pump while it is not needed. We want to avoid unnecessary loss of methylene chloride from evaporation.

8. Using forceps, transfer the filter and filter cake back into the original blender canister. Extract (blend) for 30 seconds with 200 mL of methylene chloride: methanol, 2:1 (v/v).

9. Vacuum filter the second extract through a second glass fiber filter in the same funnel, into the same flask as the original extract.

10. Release the canister for washing. Discard the final filter cake in a container for soft, contaminated waste.

B. EXTRACT WASHING PROCEDURE AND LIPID DETERMINATION:

1. Transfer the extract from the flask to a separatory funnel as completely as possible. Rinse the flask with 185 mL of water. Transfer the water rinse to the separatory funnel with the extract.

2. GENTLY mix the water with the methylene chloride: methanol by inverting the separatory funnel 20 times. Do not shake the funnel.

3. Let the funnel stand in a ring support for at least 10 minutes while the two phases separate. In some cases it may be necessary to encourage complete separation by introducing a glass rod and stirring the lower phase.

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EXTRACTION OF BIOLOGICAL MATERIAL USING WARING BLENDER	
TLI SOP No. DSP293 Version: 1	Date Revised: 18 November 1999

4. When the phases have clarified, drain the lower (methylene chloride) phase through a glass wool plugged funnel containing anhydrous sodium sulfate, into a **pre-weighed**, clean 500 mL flat bottom flask. Rinse the sodium sulfate with 30 mL of methylene chloride into the flask.

5. Rotary evaporate and exchange with n-Heptane. DO NOT ADD TRIDECANE! DO NOT ADD CLEANUP STANDARD YET!

6. Continue rotary evaporation until no more solvent will come off. Let the flask come to room temperature, wipe off all moisture from the outside of the flask with a Chem Wipe, and re-weigh the flask. This new weight minus the original flask weight, is the weight of lipid. The weight of lipid, divided by the weight of sample extracted, is the lipid content per g of sample, fresh weight basis.

7. If the weight of lipid is less than 0.5 g, AND the project required lipid determination, a more accurate weighing will be required. It will be necessary to transfer the extract, using three 2 mL rinses with methylene chloride, to a pre-weighed scintillation vial. Evaporate the solvent with a stream of nitrogen and re-weigh the vial. The final weight minus the initial weight is the weight of lipid.

NOTE 5: Use a balance capable of reading to three decimal places with the flask, or one capable of reading to four decimal places with the scintillation vial.

8. Add cleanup standard to the flask, rinsing it down with a small amount of n-Heptane. Transfer the flask for cleanup as usual for PCDD/PCDF or PCB analysis.

Comment: The initial estimation of lipid weight in the evaporation flask is made whether lipid content is to be reported or not. This measurement is used to guide the subsequent cleanup. If the lipid weight is over 5 g, the higher of the two options for the amount of acid-coated silicic acid in the SOP on Alternative Acid Cleanup should be used.

V. REFERENCE:

Albro, P.W., Schroeder, J.L., and J.T.Corbett. Lipids, 27: 136-143 (1992)

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DETERMINATION OF LIPID CONTENT FOR TISSUE EXTRACTS

TLI SOP No. DSP286	Version: 1	Effective Date:	June 12, 1998
Author:	Phil Albro	Date Written:	June 11, 1998
Authorization:	Phil Albro Management	Date Authorized:	6/11/98

I. **SCOPE AND APPLICATION:** This SOP describes one method for lipid determination in extracts from tissues intended for PCDD/PCDF analysis. It can be used when the sample size is adequate to provide at least 0.5 g of lipid in the extract, and is primarily intended for samples that will be extracted using Soxhlet or Dean-Stark extractors.

II. **SAFETY CONSIDERATIONS:** Wear lab coat, gloves and safety glasses. Perform operations under a hood. Samples may contain toxic substances. The organic solvents may be flammable and/or cause respiratory distress. For additional safety information, see TLI Safety and Health Manual and the appropriate MSDS.

III. **HEALTH FACTOR:** If the procedure described in this SOP is applied to samples of human tissue of any kind (not only blood), including urine or other human wastes, the "Universal Precautions" as recommended by the Center for Disease Control must be followed, and all employees working with this material must have received training on OSHA's BloodBorne Pathogen regulation.

IV. **PROCEDURE:**

A. Equipment and Glassware:

1. 500 mL flat-bottom flasks, one for each sample

2. Top Loading balance capable of weighing to 0.01 g.

3. Rotary Evaporator.

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DETERMINATION OF LIPID CONTENT FOR TISSUE EXTRACTS	
TLI SOP No. DSP286 Version: 1	Date Written: June 11, 1998

B. Materials:

1. Methylene chloride, Pesticide Grade or better

2. Anhydrous Sodium Sulfate, Pesticide Grade

C. Sample Processing

1. Pre-weigh 500 mL flatbottom flasks, one for each sample, to the nearest 0.01 g. These are the flasks in which the sample extract will be collected, e.g. the flasks used on a Soxhlet extractor.

2. Perform the extraction.

3. Examine the extracts. If there is solid (insoluble) residue, or visible water (two liquid phases), another pre-weighed flask will be needed. Pass the extract through a glass wool-plugged funnel containing about 10 g of anhydrous sodium sulfate, into the pre-weighed flask. Rinse the original flask with 10 mL of whatever solvent was used originally, and pass through the sodium sulfate into the second flask.

4. Rotary evaporate all samples (bath temperature at 40 degrees C). Continue the evaporation until no more solvent is condensing in the trap, but do not try to take the residue to dryness.

5. Add 10 mL of methylene chloride to the residue in the flask and repeat step 4.

6. Remove the flask from the rotary evaporator. Wipe off the outside of the flask with a Chim Wipe, cover with aluminum foil, and let it cool to room temperature.

7. Remove the foil, weigh the flask and contents to the nearest 0.01 g.

8. Continue with processing (addition of cleanup standard if appropriate, solvent exchange to heptane, etc.) as per the SOP being followed.

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DETERMINATION OF LIPID CONTENT FOR TISSUE EXTRACTS	
TLI SOP No. DSP286 Version: 1	Date Written: June 11, 1998

Calculate percent lipid as follows:

$$\% \text{ LIPID} = \frac{[\text{Wt. flask + residue, step C.7.}) - (\text{Wt. flask, step C.1 or C.3.})] \times 100\%}{\text{Original sample weight}}$$

NOTE 1: The objective is to weigh the lipid, not residual solvent or water droplets. Step 5 above helps eliminate residual toluene and also co-distills very small amounts of water.

NOTE 2: A container weighs "light" if it is above ambient temperature when put on the balance. That is the reason for waiting in step 6. above.

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CONCENTRATION OF EXTRACTS USING ROTARY EVAPORATION	
TLI SOP No. DSP124 Version: 15	Date Revised: April 19, 2000

Author: Kenneth Carroll

Effective Date: 24 April 2000

Authorization:* Phil Albro
Management

Date Authorized: 18 April 2000

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I. PURPOSE: The purpose of this SOP is to describe the procedures for the concentration of extracts and intermediate fractions by rotary evaporation.

II. SCOPE: This procedure is to be carried out by laboratory personnel trained in the use of the rotary evaporators for the concentration of samples before chromatographic cleanup. These procedures are applicable to extracts for PCB and PCDD/PCDF analysis, as well as to fractions from various cleanup steps.

III. SAFETY CONSIDERATIONS: Conduct these operations under a hood. Wear labcoat, appropriate eyewear, and gloves. These solvents are flammable; avoid flames and sparks. Do not breathe the vapors, and avoid contact with skin and eyes. They may cause irritation, nausea, dizziness, and, in extreme cases, death. For additional safety and health information, see the TLI Safety and Health Manual and the appropriate MSDS.

IV. PROCEDURE:

A. CHECK INSTRUCTIONS IN FOLDER:

1. Check the folder to determine whether a given sample is for PCDD/PCDF or PCB analysis, whether or not the extract will be split among multiple assays, whether or not there are special spiking instructions, whether or not a portion of the extract will be removed for lipid determination, and whether or not this sample is from partial cleanup and therefore doesn't need solvent exchange.

2. Note that if this extract will be used for PCB analysis, no tridecane is added. If the extract will be split between PCDD/PCDF and PCB analysis, again no tridecane is added.

3. If the sample is an extract of animal tissue or a QC sample containing corn oil, no tridecane is needed. The lipid serves the same function.

CONCENTRATION OF EXTRACTS USING ROTARY EVAPORATION	
TLI SOP No. DSP124 Version: 15	Date Revised: April 19, 2000

4. Determine what, if any, "cleanup standard" is to be added to the extracts, and at what point in the concentration process it is to be added.

B. DISMANTLE SOXHLETS:

1. It may be necessary to take down the Soxhlet extractors before concentrating extracts.

2. After samples have cooled , remove thimble holder (also remove SDS-Soxhlet Dean Stark extractor). Drain water from the SDS in the water waste, drain the Toluene or other non-chlorinated solvent into the non-chlorinated waste and the methylene chloride into the methylene chloride waste. Place glassware in a bucket to be taken to the washroom. No more than one sample's glassware should be uncovered at any given time.

3. Cover samples with foil and move them to the rotovap area.

C. ADDITION OF TRIDECANE (PCDD/PCDF ANALYSIS ONLY):

NOTE: Pipette tips must be changed for each sample. Fresh tridecane must be aliquoted every day into a new scintillation vial. At the end of the day, dispose of any remaining tridecane, and throw away the scintillation vial in a sharps contaminated waste container.

1. For routine 8290 or 1613 Pulp, Paper, Cardboard, Ash, Soil, Water, Sludge, and Sediment samples, 500 µL of tridecane is added, then the samples must be concentrated to 0.5mL.

2. For GP 8290 and 551 Pulp, Paper, Cardboard, Ash, Soil, Water, Sludge, and Sediment samples, the sample must be concentrated to 10 mL, then add 500 µL of Tridecane and 40 mL of Heptane before rotary evaporation to 0.5 mL.

3. For 8290, 1613, and 551 Fish and Other Tissue samples, 5 mL must be removed for % Lipid determination, then samples must be concentrated and solvent exchanged. For methods 8290, 1613, and 551, add tridecane to the OPR, TLI Blank, and LCS/LCSD unless corn oil was used as matrix for QC samples.

4. For 8280, DFLM01.1 and 613 solid samples, 500µl of Tridecane is added before rotary evaporation.

5. Note that tridecane is not added if the extracts will be split between PCDD/PCDF and PCB analysis. Instead, evaporation must be carefully

watched, stopped when about 3 mL of solvent remains, and followed by solvent exchange with heptane or isooctane.

D. **COMBINING EXTRACTS**

1. If water samples are filtered, there will be both a methylene chloride extract of the filtrate and a toluene extract of the filter. All samples in these projects will have a methylene chloride extract, but only those samples that were filtered will have a toluene extract. Therefore, for consistency, the tridecane (if used) and cleanup standard (if used) should be added to the methylene chloride extract, and it should be concentrated first.

2. Once the methylene chloride extract has been concentrated, EITHER perform solvent exchange with heptane if there is no toluene portion, OR add the toluene extract (if there is one) to the flask containing the residue from the methylene chloride extract. Rinse the toluene extract flask with 5 mL of toluene, swirl to rinse the entire inside of the flask, and transfer the rinse to the other flask. Continue with concentration, followed by solvent exchange.

E. HOW TO CLEAN AND OPERATE THE ROTARY EVAPORATOR:

1. Cleaning Procedure:

   The following steps should occur in the cleaning of the shafts and traps. First, each day you should start with clean shafts and traps. Once you start rotary evaporating, the following solvents are needed for cleaning and flushing shafts, traps and condensers after each sample and for flushing after rotary evaporating highly contaminated samples. The shafts and traps should be rinsed in the following order:

   a) Acetone

   b) Methanol

   c) Methylene Chloride

   d) Heptane

      *If a sample(s) is suspected to be highly contaminated, after the samples have been evaporated, the following method is used to decontaminate the system.

   e) Increase temperature of water bath to 40-45°C before rotary evaporating.

Note: Be careful not to exceed 45°C; if so, document in rotary evaporation logbook.

f) To flush the system, use a 250 mL round bottom flask to rotary evaporate 100 mL of each of the four solvents above.

g) Remove the shaft and small trap from the rotary evaporator, remove all vacuum grease with methylene chloride, and take them to the washroom for cleaning.

2. Operation:

Daily routine check: before operating the rotary evaporator, do the following:

a) Make sure that the shaft and small trap have been replaced with a clean one. (A clean shaft and trap is indicated by a piece of aluminum foil covering the trap opening). Shafts and traps must be replaced after each batched project.

b) If in doubt, remove the shaft and small trap from the rotary evaporator, degrease the shaft with paper towel dampened with methylene chloride. Wrap the shafts in aluminum foil and take to the washroom for cleaning, then change latex gloves.

c) Obtain a cleaned set-up from the washroom. Rinse with acetone, methanol, methylene chloride, and heptane. Drain.

d) Before installing the clean shaft, grease the ground glass area by applying a small amount of vacuum grease. Insert the shaft and twist to ensure proper sealing. If any grease oozes out on the assembly, there is too much grease. Take a paper towel and wipe off the excess grease. Then change latex gloves.

e) Attach the small trap to the shaft with a clamp (no grease). Cover the opening with aluminum foil.

f) Fill water bath between one-half to two-thirds full with deionized water as needed during the use of the rotary evaporator.

g) Empty the cold trap if it is 2/3 full of solvent.

h) Set the water bath temperature control knob to the desired temperature, increasing or decreasing by small increments.

i) The appropriate temperature for rotary evaporating the following solvents: NOT to exceed 45°C.

(1)	toluene	40 to 44°C
(2)	methylene chloride	35 to 40°C
(3)	iso-octane	40 to 44°C
(4)	ethanol	40 to 42°C

j) Turn on the refrigerated circulator. If the coolant is low in volume, add a mixture of methanol/deionized water 1:1. The desired temperature range is 1 to 10°C.

k) Adjust the speed control knob to zero.

l) Check the glass stopcock at the top of the condenser and apply vacuum grease if needed.

3. Rotary Evaporating Sample:

a) First check to see if all samples were spiked with all standards and if Tridecane was added (if applicable).

b) Attach the sample flask to the small trap with a clamp (no grease). Do this only after the desired temperature of the coolant and water baths have been reached.

c) Turn on the vacuum pump with the glass stopcock open. Start with 20 psi pressure setting.

d) Close stopcock and turn the speed control knob to a slow speed.

e) **Do Not immediately lower flask into the water bath**, let the flask rotate for 5-10 minutes. Time may vary according to amount of liquid in sample.

f) Press the bar on the rotary evaporator and lower the flask until it touches the water in the water bath.

g) Adjust flask submersion depth and rotation for maximized distilling.

h) (If signs of bumping occur, immediately release the vacuum, raise the flask from the bath, and decrease rotation speed).

i) Start again by closing the condenser stopcock. Lower the flask by touching the water and gradually increase the depth and rotation speed. If signs of bumping no longer occur, gradually increase the pressure by turning the valve on the pump clockwise. (DO NOT leave sample unattended until all signs of bumping disappears).

j) If bumping occurs, (when the sample in the flask shoots up in the small trap and shaft) do not return the sample trap back in to the sample flask. Document in the project folder and in MILES if sample volume has been lost. Document bumping in the rotovap run log, initial and date to close entry.

k) Rinse shaft and trap with acetone, methanol, methylene chloride, and heptane.

l) Repeat Step i.

m) Check the Sample Extraction and Clean-up Tracking Form for instructions regarding the final volume of the extract.

n) When the conditions of the instructions have been met, stop rotation and raise the flask above water level.

o) Open the glass valve on top of the condenser to release the vacuum; then turn off the vacuum pump.

p) When the pressure equalizes, remove the flask. (DO NOT force the flask from the trap, wait until the vacuum is released).

Note: If the sample has a combine portion, it should be combined and rotary evaporated before it is solvent exchanged.

q) Solvent exchange with 20 mL heptane twice all samples whose next step is chromatographic cleanup. Concentrate to 0.5 mL each time to remove any residual solvent unless tridecane is not being used, in which case concentrate to 3 mL each time.

Note: For Isolation Samples (Method 8280 and 613), see section C.4.

r) Remove the small trap and rinse with the solvents as in step 11.

s) Proceed with the next sample.

CONCENTRATION OF EXTRACTS USING ROTARY EVAPORATION	
TLI SOP No. DSP124 Version: 15	Date Revised: April 19, 2000

Note: Do not mix solvents in the waste trap. Empty the trap into the appropriate waste bottle before evaporating a sample with a different solvent.

4. For Isolation samples (Method 8280 and 613)

   a) Add 10 mL of heptane to the sample flask and sonicate well.

   b) Transfer the extract to a scintillation vial calibrated to 20 mL. Rinse the sample flask twice with 5 mL of heptane, add rinses to the vial.

   c) Adjust volume in the vial to exactly 20 mL, mix well, and then immediately transfer required portion (usually 20% - 4 mL) back to the original flask. Mark the original flask with "20%" and cover flask with foil. If the sample will not undergo cleanup procedures within 12 hours, the flask must be covered with Teflon tape before covering with aluminum foil.

   d) Spike the sample portion in the flask with 100 μL of 8280 Surrogate Standards at 0.05μg/mL, and relinquish to cleanup room. For method 613, spike the sample with 20μL of 613 surrogate standard at 0.014 μg/mL.

   e) Mark the level of the sample extract remaining in the scintillation vial and label "80%" and solvent used (heptane) for solvent exchange. Archive the sample extract in the cooler #2 after recording the appropriate information into isolation Archive Logbook.

   f) Remove the small trap and rinse with the solvents as in step 11.

   g) Proceed with next sample.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290			
TLI SOP No. DHR182	Version: 6	Effective Date:	March 25, 1998
Author:	Wojciech Krol	Date Written:	February 9, 1998
Authorization:	[signature] Management	Date Authorized:	2/16/98

I. SCOPE AND APPLICATION: This method provides procedures for the analysis of polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologues; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologues; PCDFs) from extracts of samples prepared according to SW 846 Method 8290.

II. SAFETY CONSIDERATIONS: The 2,3,7,8-TCDD isomer has been found to be acenegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in the 2,3,7,8 positions are known to have toxicities comparable to that of 2,3,7,8-TCDD. Extreme care must be exercised in all handling of extracts and standards in the application of this SOP. For additional safety information, see the TLI Safety and Health Manual and the appropriate MSDSs.

III. EQUIPMENT: The High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (GC/MS/DS) equipment utilized for this analysis includes the following:

A. Gas Chromatographs: Hewlett Packard 5890 or 5890 Series II (equipped for temperature programming and capillary columns)

B. High Resolution Mass Spectrometers: VG 70 Series, VG 70-SE Series, VG Autospec.

C. Data Systems: VG Analytical PDP11 with 11-250 software or VAX Alpha with Opus 3.2 software.

D. GC Injection Port - The GC injection port is designed for capillary columns. Typically, 2 μL injection volumes are used unless otherwise noted.

E. Gas Chromatograph/Mass Spectrometer(GC/MS) Interface - The GC/MS interface components can withstand 350°C. The interface has been designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. The GC column is fitted directly into the mass spectrometer ion source without being

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exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel™, or equivalent, ferrules are recommended.

F. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley).

G. Data System - A dedicated data system is employed to control the rapid selected-ion monitoring (SIM) process and to acquire the data. Quantitation data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantitation may be reported based upon computer generated peak areas or upon measured peak heights. The data system is set to acquire data as low as 10 ions in a single scan. Table 1. presents a listing of the ions which are typically monitored. The data system is set to switch to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system provides hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It also acquires mass spectral peak profiles and provides hard copies of peak profiles to demonstrate the required resolving power. Measurements of noise on the base line are performed using the hard copies of individual ion chromatograms provided by the data system.

NOTE: The detector ADC zero setting must be set to allow peak-to-peak measurement of the noise on the base line of every monitored channel and allow for good estimation of the instrument resolving power.

H. GC Columns

1. In order to have an isomer specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60 m DB-5 fused silica capillary column is used.

2. The 2,3,7,8-TCDF isomer must be confirmed on a 30 m DB-225 fused silica capillary column, when 2,3,7,8-TCDF is detected on the DB-5 column at a level greater than or equal to the target detection limit.

IV. **STANDARDS:**

A. Calibration Solutions Six nonane solutions containing 17 unlabeled PCDDs and PCDFs and 18 $^{13}C_{12}$ -labeled PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The analyte concentration ranges are homologue dependent, with the lowest values for the tetrachlorinated dioxin and

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furan (0.5 pg/μL) and the highest values for the octachlorinated congeners (2000 pg/μL) (Table 2.).

B. Recovery Standard Solution - This nonane solution contains two recovery standards, $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD, at a nominal concentration of 100 pg/μL per compound. A 20 μL of this solution is spiked into each sample extract before the HRGC/HRMS analysis.

C. GC Column Performance/Retention Window Check Solution (RTCHK)- This solution contains the first and last eluting isomers for each homologous series from tetra- through heptachlorinated congeners. The solution also contains a series of closely eluting TCDD and TCDF isomers for the purpose of documenting the chromatographic resolution (Table 3).

D. Acceptance Criteria for Newly Prepared Standards - All components and concentrations of each calibration standard, recovery standard, internal standard, and matrix spike solutions are verified prior to use for the analysis of samples. Testing consists of back to back analysis of the "test" solution (the newly prepared solution) and a "control" solution (a known good solution). Control solutions are isolated from the production standards in a protected location. Both the control and test solutions are evaluated versus the current continuing calibration standard and versus each other. Each component of the test solution must be within 80 - 120% of the true concentration when calculated versus the control standard. The control standard must be within 80 - 120% of the true value when calculated versus the continuing calibration standard.

E. Standards are stored in 1/2 dram amber glass vials at room temperature.

V. SYSTEM PERFORMANCE CRITERIA

System performance criteria are presented below. It must be documented that all applicable system performance criteria specified in this Section are met before analysis for any sample is performed. Table 4 provides recommended GC conditions that can be used to satisfy the required criteria. During a typical 12-hour analysis sequence, the GC column performance and mass spectrometer resolving power checks must be performed at the beginning of the 12-hour period of operation. A routine calibration verification is required at the beginning and end of each 12-hour period during which samples are analyzed. A method blank or HRGC/HRMS solvent blank is required between a calibration run and the first sample run.

A. GC Column Performance

1. Inject the column performance check solution (Section IV. C.) and acquire selected-ion monitoring (SIM) data as described in Section III. E., III. F., and Table 4.

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2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers or between 2,3,7,8-TCDF and the peaks representing other TCDF isomers must be resolved with a valley of $\leq$ 25% (Fig. 1.), where:

   Valley Percent = (x/y) x 100

   x = height of valley measured between 2,3,7,8-TCDD or 2,3,7,8-TCDF and the closest TCDD or TCDF eluting isomers, and

   y = the peak height of 2,3,7,8-TCDD or 2,3,7,8-TCDF.

3. The acquisition time windows must be set to allow observation of the first and last eluting isomer of each congener. All first and last eluters of a homologous series should be labeled and identified on chromatograms.

B. Mass Spectrometer Performance

1. The mass spectrometer must be operated in the electron ionization mode. It is recommended that the ionization potential be set to optimize sensitivity for the given column flow and source design. A static resolving power of at least 10,000 (10% valley definition) must be demonstrated at appropriate masses before any analyses are performed. Static resolving power checks must be performed at the beginning and at the end of each 12-hour period of operation. It is recommended, however, that a check of the static resolution be made and documented by using the peak matching unit before and after each analysis. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, use a lock-mass ion from the reference compound Perfluorokerosene (PFK) used for tuning the mass spectrometer and monitor and record the lock-mass ion channel during SIM acquisitions. The level of the reference compound (PFK) metered inside the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the selected lock-mass ion signal, regardless of the description number, does not exceed 10% of the full scale deflection for a given set of detector parameters. Under those

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conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

3. Using PFK molecular leak and an appropriate ion within the scan window, tune the instrument to meet the minimum required resolving power of 10,000 (10% valley).

    a) Documentation of the mass spectrometer resolving power is accomplished by recording the peak profile of the high-mass reference signal (m/z 416.9760) obtained during a peak matching experiment by using the low-mass PFK ion at m/z 330.9792 (or lower in mass) as a reference.

    b) The format of the peak profile representative allows manual determination of the peak resolution. The peak width (at 5% peak height) of the high-mass reference ion must not exceed 100 ppm (resolving power: 10,000). Peak width is determined by triangulation with no more than 10% allowance for sampling error. Instrumental ion transmission and resolution will be checked, adjusted and documented in case the resolution is below the minimum required 10,000 resolving power.

VI. CALIBRATION PROCEDURES:

A. Initial Calibration (ICAL)- Initial calibration of the instrument is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any continuing calibration does not meet the required criteria listed in Section VI. B..

1. All six calibration solutions listed in Table 2. must be used for the initial calibration.

2. Tune the instrument with PFK to achieve a static resolving power of at least 10,000 (10% valley) as described in section V. B..

3. Inject 2 µL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section III. F. - G. Any further analysis must not be performed until it has been documented that the column performance criteria listed in section V. A. was met.

4. Using the same GC and MS conditions that produced acceptable results for the column performance check solution, analyze a 2 µL portion of each of the six calibration solutions with the following requirements.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

a) The ratio of integrated ion current for the ions appearing in Table 5 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series).

b) The ratio of integrated ion current for the ions belonging to the carbon-labeled internal, surrogate, alternate, and recovery standards must be within the control limits stipulated in Table 5.

NOTE: Ion ratios for all 17 native analytes and 18 carbon-labeled internal and recovery standards must be within the specified control limits simultaneously in one run for each of the six (6) calibration standard solutions. If the ion abundance ratios are outside the limits, corrective action must be taken and acceptable abundance ratios achieved before any samples may be analyzed.

c) For each SICP and for each GC signal representing the elution of a target analyte the signal-to-noise ratio (S/N) must be better than or equal to 10:1.

d) Referring to Table 6., calculate the 17 relative response factors (RRF) for unlabeled target analytes relative to their appropriate internal standards, according to the following formula:

$$RRF(n) = \frac{A_x \times Q_{is}}{Q_x \times A_{is}}$$

Where:

RRF(n)=		Analyte RRF
A_x	=	sum of the integrated ion abundances of the quantitation ions (Table 1.) for unlabeled PCDDs/PCDFs,
A_{is}	=	sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standards,
Q_{is}	=	quantity of the internal standard injected (pg),
Q_x	=	quantity of the unlabeled PCDD/PCDF analyte injected (pg).

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The RRF(n) is a dimensionless quantity; the units used to express Q_{is} and Q_x must be the same.

e) Calculate the average analyte $\overline{RRF}$ and their respective percent relative standard deviations (%RSD) for the six calibration solutions.

$$\overline{RRF}_{(n)} = \frac{1}{6}\sum_{j=1}^{5} RRF_{nj}$$

Where n represents a particular PCDD/PCDF (2,3,7,8 substituted congener), and j is the injection number (or calibration solution number).

f) The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

(1) For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section V. A. 4. e).

NOTE: The calibration solutions do not contain $^{13}C_{12}$-OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the $[M+6]^+$ ion of $^{13}C_{12}$-OCDF from the $[M+2]^+$ ion of OCDD (and $[M+4]^+$ from $^{13}C_{12}$-OCDF with $[M]^+$ of OCDD). Therefore, the RRF for OCDF is calculated relative to $^{13}C_{12}$-OCDD.

(2) For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (PeCDF - two, HxCDF - four, HxCDD - three, HpCDF - two) the mean RRF used for those homologous series will be the average of the mean RRFs calculated for all individual 2,3,7,8-substituted congeners.

NOTE: HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution pattern are assumed to be the same as the

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

g) Referring to Table 7. and 8. calculate relative response factors (RRF) and average relative response factors (RRF) for internal, surrogate, and alternate standards relative to their appropriate recovery standards, according to the following formula:

$$RRF_{(m)} = \frac{A_s \times Q_{rs}}{Q_s \times A_{rs}}$$

$$\overline{RRF}_{(m)} = \frac{1}{6} \sum_{j=1}^{6} RRF_{(m)j}$$

Where:

m = congener type,

j = injection number,

A_s = sum of the integrated ion abundances of the quantitation ions for a given standard,

A_{rs} = sum of the integrated ion abundances of the quantitation ions for the appropriate recovery standard,

Q_{rs}, Q_s = quantities of, respectively, the recovery standard (rs) and a particular standard injected (pg),

$RRF_{(m)}$ = relative response factor of a particular standard relative to an appropriate recovery standard, as determined from one injection, and

$\overline{RRF}_{(m)}$ = calculated mean relative response factor of a particular labeled standard relative to an appropriate recovery standard, as determined from the six initial calibration injections.

5. Acceptance Criteria for Initial Calibration - The criteria listed below for acceptable calibration must be met before any analysis is performed.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

a) The percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 20 percent, and those for the labeled reference compounds must not exceed ± 30%.

b) The S/N ratio for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

c) The isotopic ratios (Table 5.) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section VI. A. 5. a) is met, the analyte specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the continuing calibration criteria (Section VI. B.) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

B. Continuing Calibration Check (CONCAL) - Continuing calibrations must be performed at the beginning of a 12 hour period after successful mass resolution and QC resolution performance checks. A continuing calibration is also required at the end of a 12 hour shift.

1. Inject 2 µL of the CONCAL solution HRCC-3 standard (Table 2). By using the same HRGC/HRMS conditions as described in Section V., and used to determine and document an acceptable calibration as provided in Section VI. A. 5..

2. Acceptance Criteria for Continuing Calibration - The following criteria must be met before further analysis is performed.

a) The measured RRFs for the unlabeled standards obtained during the continuing calibration runs must be within ± 20 percent of the mean values established during the initial calibration (Section VI. A. 4. e) and f)).

b) The measured RRFs for the labeled standards obtained during the routine calibration must be within ± 30 percent of the mean values established during the initial calibration (Section VI. A. 4. g)).

c) The ion-abundance ratios (Table 5.) must be within the allowed control limits.

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d) If either one of the criteria in Sections VI. B. 2. a) and b) is not satisfied, repeat one more time. If these criteria are still not satisfied, the entire continuing calibration process must be reviewed.

e) If the continuing calibration at the end of a 12-hour period fails no more than 25% RPD for the unlabeled compounds and 35% RPD for the labeled reference compounds, use the mean RRFs from the two daily calibration runs to compute analyte concentrations, instead of the RRFs obtained from the initial calibration.

NOTE: If RRFs for up to two labeled standards fail by more than 35%, the calibration is considered acceptable, as long as corresponding unlabeled analytes meet criteria.

f) Continuing calibrations analyzed at the end of the 12 hour period which fail by more than 25% RPD for the unlabeled compounds and 35% RPD for the labeled reference compounds must be documented according to the non-conformance SOP. The analyst must assess the effect of failure on overall data quality. The assessment will include evaluation of any spiked samples included in the 12 hour "clock", presence of reportable levels of the affected analytes in the samples, and sample data obtained during the period between beginning and ending calibrations. All samples which contain reportable levels of a "failed" analyte will be reanalyzed unless a passing spiked sample has been analyzed between the sample and the ending calibration standard.

VII. ANALYSIS

A. A valid analysis of column performance check, calibration and method or instrument blank must have been obtained prior to the analysis of any sample.

B. Remove the sample extract (blown to dryness) or blank from storage. Add 20 µL of recovery standard and mix thoroughly. Care must be taken to coat the walls of the vial several times to dissolve the sample material deposited on the walls during the concentration process.

NOTE: A final volume of 20 µL or more should be used whenever possible. A 10 µL final volume is difficult to handle, and injection of 2 µL out of 10 µL leaves little sample for confirmations and repeat injections, and for archiving.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

C. Inject a 2 µL aliquot of the extract into the GC, operated under the GC conditions that have produced acceptable CONCAL and RTCHK results.

D. Acquire SIM data using the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors.

NOTE: The acquisition period must at least encompass the PCDD/PCDF overall retention time window previously determined. Selected ion current profiles (SICP) for the lock-mass ions (one per mass descriptor) must also be recorded and included in the data package. These SICPs must be true representations of the evolution of the lock-mass ions amplitudes during the HRGC/HRMS run. The analyst may be required to monitor a PFK ion, not as a lock mass, but as a QC ion, in order to meet this requirement. It is recommended to examine the QC ion or lock-mass ion SICP for obvious basic sensitivity and stability changes of the instrument during the GC/MS run that could affect the measurements. Report any discrepancies in the case narrative.

E. Identification Criteria - For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

1. Retention Times

a) For 2,3,7,8-substituted congeners, which have an isotopically labeled internal or recovery standard present in the sample extract (this represents a total of 10 congeners including OCDD; Table 1.), the retention time (RRT; at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 1.), must be within -1 to +3 seconds of the isotopically labeled standard.

b) For 2,3,7,8-substituted compounds that do not have an isotopically labeled internal standard present in the sample extract (this represents a total of eight congeners; Table 1.), the retention time must fall within 0.005 retention time units of the relative retention times measured in the continuing calibration. Identification of OCDF is based on its retention time relative to $^{13}C_{12}$-OCDD as determined from the 12 hour continuing calibration results.

c) For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the RTCHK solution.

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d) The ion current responses for both ions used in quantitation (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach maximum simultaneously (± 2 seconds).

e) The ion current responses for both ions used for the labeled standards (e.g., for $^{13}C_{12}$-TCDD: m/z 331.9368 and m/z 333.9339) must reach maximum simultaneously (± 2 seconds).

2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned (Table 5.).

3. Signal-to-Noise (S/N) Ratio

All ion current intensities must be ≥ 2.5 times noise level for positive identification of a PCDD/PCDF compound or a group of coeluting isomers.

4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N ≥ 2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 1.) channel or if the PCDPE signal is less than 10% of the PCDF signal.

F. Quantitation

1. For gas chromatographic peaks that have met specified criteria calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times \overline{RRF}_{(n)}}$$

Where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) in pg/g,

A_x = sum of the integrated ion abundances of the quantitation ions (Table 1.) for unlabeled PCDDs/PCDFs,

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A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standards,

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

W = weight or volume, in grams or liters, of the sample (solid or liquid), and

$\overline{RRF}_{(n)}$ = calculated mean relative response factor for the analyte.

2. Calculate the percent recovery of the nine internal standards measured in the sample extract. using the formula:

$$\text{Internal Standard Recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times \overline{RRF}_{(m)}} \times 100$$

Where:

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standard,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (Table 7.),

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

Q_{rs} = quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$\overline{RRF}_{(m)}$ = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 7) recovery standard.

3. Calculate the percent recovery of seven surrogate and alternate standards using expression VII. F. 2..

NOTE: For the air samples (PUF, M-0023A) calculate percent recovery of five surrogate standards relative to their appropriate internal standards.

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4. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compound exceeds the dynamic range of the instrument (i.e., saturation in a mass channel) then solvent will be added to the extract to bring the signal level into the instrument's dynamic range. Any dilutions must be pre-approved by the client.

5. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value must be specified in the report.

6. Sample Specific Estimated Detection Limit - The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, depending on the type of response produced during the analysis of a particular sample.

   a) Samples giving a response for at least one quantitation ion that is less than 2.5 times the background level.

      The area of the analyte is replaced by the noise level measured in a region of the chromatogram clear of genuine GC signals multiplied by an empirically determined factor. The detection limits represent the maximum possible concentration of a target analyte that could be present without being detected.

$$DL = \frac{2 \times 2.5 \times (F \times H) \times Q_{is}}{A_{is} \times \overline{RRF}_{(n)} \times W}$$

Where:

DL	=	estimated detection limit for a target analyte, expressed in ng or pg.
2.5	=	minimum signal/ noise required for a GC signal to be accepted.
F	=	an empirical number that approximates the area to height ratio for a GC signal. (F= 3.7 for all dioxin/ furan analyses.)

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

H	=	height of the noise.
A_{is}	=	integrated current of the characteristic ions of the corresponding internal standard.
Q_{is}	=	amount of internal standard added to the sample before extraction.
$\overline{RRF}_{(n)}$	=	mean analyte relative response factor from the initial calibration.
W	=	sample weight or volume.
2	=	used to account for the two signals present for every analyte.

b) Samples characterized by a response above the background level with a S/N of at least 2.5 for both quantitation ions.

When the response of a signal having the same retention time as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet ion ratio requirements (Table 5.) calculate the "Estimated Maximum Possible Concentration" (EMPC) according to the expression shown in Section VII. F. 1., except that A_x should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio.

VIII. QUALITY CONTROL REQUIREMENTS

A. GC column performance must be demonstrated initially and verified prior to analyzing any sample in a 12-hour period. The GC column performance check solution must be analyzed under the same chromatographic and mass spectrometric conditions used for other samples and standards.

B. Routine calibrations must be performed at the beginning of a 12-hour period after successful mass resolution and GC resolution performance checks and at the end of a 12-hour period following the analysis of samples.

C. The target detection limits will be the following:

1. TDLs for waters (sample size - 1L):

$\leq$ 10 ppq	TCDD & TCDF
$\leq$ 50 ppq	Penta - HeptaCDD & CDF
$\leq$ 100 ppq	OCDD & OCDF

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

2. TDLs for solids (sample size - 10g):

   ≤ 1 ppt TCDD & TCDF

   ≤ 5 ppt Penta - HeptaCDD & CDF

   ≤ 10 ppt OCDD & OCDF

3. For air (e.g., PUF, M-0023A) or Wipes (sample size - 1):

   ≤ 50 pg TCDD & TCDF

   ≤ 250 pg Penta - HeptaCDD & CDF

   ≤ 500 pg OCDD & OCDF

D. Method Blank

1. Percent recoveries of all labeled standards should be between 40 - 135%. Method blank with standard recoveries below 40% (but ≥ 25%) is acceptable as long as signal to noise for affected standards is ≥ 10:1.
2. Target detection limits must be met for each analyte. (VIII. C.)
3. No reportable analytes should be present in the Method Blank. Up to 3 analytes may be present at levels below 1/2 Target Detection Limit (TDL) as long as the compounds were not reported in the previous analysis.
4. For Method Blanks associated with high level samples, the analyte level in the blank must be < 5% of the quantity present in the samples.

E. LCS/ LCSD and MS/ MSD

1. Percent recoveries of all labeled standards should be between 40 - 135%. LCS/ LCSD or MS/ MSD with labeled standard recoveries below 40% (but ≥ 25%) are acceptable as long as signal to noise for affected standards is ≥ 10:1.
2. Percent recoveries of all analytes should be between 70 - 130%. For up to two analytes, recoveries may be as high as 145% or as low as 60%, as long as the associated relative percent differences (%RPDs) meet criteria.
3. Relative percent difference between LCS and LCSD or MS and MSD should be ≤ 20% for all analytes. For up to two analytes, %RPDs may be higher (up to 35%), as long as the associated percent recoveries meet criteria.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

F. Samples

1. Percent recoveries of all labeled standards should be between 40 - 135%. Samples with standard recoveries below 40% (but $\geq$ 25%) are acceptable as long as signal to noise for affected standards is $\geq$ 10:1. In the case of OCDD internal standards, the OCDF analyte must be below TDL also.

2. Specific detection limits must not exceed the target detection limits unless prohibited by a limited sample size or the need for dilution.

3. If the method blank contains reportable analytes but those analytes are not detected in the sample, the sample data may be reported.

4. Samples which fail acceptance criteria listed above (VIII. F. 1. and 2.) or associated with failing method blank or LCS/ LCSD (VIII. D. 1. - 4.; VIII. E. 1. - 3.) must be reextracted and reanalyzed. Exceptions may be made for sample matrices which require extreme additional cleanup procedures.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 1: ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HRGC/HRMS FOR PCDDs AND PCDFs

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
2	292.9825	LOCK	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF (S)
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF (S)
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD (S)
	330.9792	QC	C_7F_{13}	PFK
	331.9368	M	$13C_{12}H_4{}^{35}Cl_4O_2$	TCDD (S)
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD (S)
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF (S)
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF (S)
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD (S)
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD (S)
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF (S)

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 1 CONTINUED

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
3 Continued	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF (S)
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	LOCK	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD (S)
	403.8529	M+4	$13C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD (S)
	445.7555	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE
	430.9729	QC	C_9F_{13}	PFK
4	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8253	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF (S)
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF (S)
	423.7766	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD (S)
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD (S)
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE
	430.9729	LOCK	C_9F_{17}	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD (S)
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD (S)

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CONFIDENTIAL and PROPRIETARY INFORMATION

PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 1 CONTINUED

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
4 Continued	513.6775	M+4	$C_{12}{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE
	442.9728	QC	$C_{10}F_{17}$	PFK

a) The following nuclidic masses were used:

H = 1.007825
C = 12.000000
^{13}C = 13.003355
F = 18.9984
O = 15.994915
^{35}Cl = 34.968853
^{37}Cl = 36.965903

S = Labeled Standard
QC = Ion Selected for Monitoring the Instrument Stability During the GC/MS Analysis

b) Descriptor 1 contains mono-, di- and trichlorinated dibenzodioxins and dibenzofurans that are not quantitated by Method 8290.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 2: COMPOSITION OF THE INITIAL CALIBRATION SOLUTIONS

Compound	Concentrations (pg/µL) Sol. Number: 1	2	3	4	5	6
Unlabeled Analytes						
2,3,7,8-TCDD	0.5	1	10	50	100	200
2,3,7,8-TCDF	0.5	1	10	50	100	200
1,2,3,7,8-PeCDD	2.5	5	50	250	500	1000
1,2,3,7,8-PeCDF	2.5	5	50	250	500	1000
2,3,4,7,8-PeCDF	2.5	5	50	250	500	1000
1,2,3,4,7,8-HxCDD	2.5	5	50	250	500	1000
1,2,3,6,7,8-HxCDD	2.5	5	50	250	500	1000
1,2,3,7,8,9-HxCDD	2.5	5	50	250	500	1000
1,2,3,4,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,6,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,7,8,9-HxCDF	2.5	5	50	250	500	1000
2,3,4,6,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,4,6,7,8-HpCDD	2.5	5	50	250	500	1000
1,2,3,4,6,7,8-HpCDF	2.5	5	50	250	500	1000
1,2,3,4,7,8,9-HpCDF	2.5	5	50	250	500	1000
OCDD	5	10	100	500	1000	2000
OCDF	5	10	100	500	1000	2000
Internal Standards						
$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100	100

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 2 CONTINUED

Compound	Concentrations (pg/µL) Sol. Number: 1	2	3	4	5	6
Internal Standards Continued						
$^{13}C_{12}$-OCDD	200	200	200	200	200	200
$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100	100
Surrogate Standards						
$^{37}Cl_4$-2,3,7,8-TCDD	0.5	1	10	50	100	200
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	100	100	100	100	100
Alternate Standard						
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100	100
Recovery Standards						
$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100	100

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 3: GC RETENTION TIME WINDOW DEFINING SOLUTION AND ISOMER SPECIFICITY TEST STANDARD (SECTION IV. C.)

DB-5 COLUMN GC RETENTION TIME WINDOW DEFINING SOLUTION

CDD/ CDF	FIRST ELUTED	LAST ELUTED
TCDF	1,3,6,8	1,2,8,9
TCDD	1,3,6,8	1,2,8,9
PeCDF	1,3,4,6,8	1,2,3,8,9
PeCDD	1,2,4,7,9	1,2,3,8,9
HxCDF	1,2,3,4,6,8	1,2,3,4,8,9
HxCDD	1,2,4,6,7,9	1,2,3,4,6,7
HpCDF	1,2,3,4,6,7,8	1,2,3,4,7,8,9
HpCDD	1,2,3,4,6,7,9	1,2,3,4,6,7,8

DB-5 COLUMN TCDD SPECIFICITY TEST STANDARD

1,2,3,7 + 1,2,3,8 - TCDD
2,3,7,8 - TCDD
1,2,3,9 - TCDD

DB-225 COLUMN TCDF ISOMER SPECIFICTY TEST STANDARD

2,3,4,7 - TCDF
2,3,7,8 - TCDF
1,2,3,9 - TCDF

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TABLE 4: GAS CHROMATOGRAPHY CONDITIONS

Column type	DB-5	DB-225
Length (m)	60	30
i.d. (mm)	0.25	0.25
Film Thickness (um)	0.25	0.25
Carrier Gas	Helium	Helium
Carrier Gas Flow (mL/min)	1-2	1-2
Injection Mode	⇐ splitless ⇒	
Valve Time (s)	60	60
Initial Temperature (°C)	150	130
Injection Port Temperature (°C)	250	250
Program Temperature	⇐ See Note ⇒	

Note: The GC temperature program is subject to change. Refer to work area guidelines for exact definitions of the current run conditions and descriptor name.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 5. ION-ABUNDANCE RATIO ACCEPTABLE RANGES FOR PCDDs AND PCDFs

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6a	M/M+2	0.51	0.43	0.59
7b	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

a) Used only for ^{13}C-HxCDF
b) Used only for ^{13}C-HpCDF

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 6. UNLABELED ANALYTES QUANTITATION RELATIONSHIPS

Analyte	Standard Used During Quantitation
2,3,7,8-TCDD	$^{13}C_{12}$-2,3,7,8-TCDD
Other TCDDs	$^{13}C_{12}$-2,3,7,8-TCDD
1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,7,8-PeCDD
Other PeCDDs	$^{13}C_{12}$-1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
Other HxCDDs	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
Other HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
OCDD	$^{13}C_{12}$-OCDD
2,3,7,8-TCDF	$^{13}C_{12}$-2,3,7,8-TCDF
Other TCDFs	$^{13}C_{12}$-2,3,7,8-TCDF
1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF
Other PeCDFs	$^{13}C_{12}$-1,2,3,7,8-PeCDF
1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 6 CONTINUED

Analyte	Standard Used During Quantitation
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
Other HxCDFs	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
Other HpCDFs	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
OCDF	$^{13}C_{12}$-OCDD

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CONFIDENTIAL and PROPRIETARY INFORMATION

PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 7: INTERNAL STANDARDS QUANTITATION RELATIONSHIPS

Internal Standard	**Standard Used During Percent Recovery Determination**
$^{13}C_{12}$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-OCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-2,3,7,8-TCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

TABLE 8: SURROGATE/ALTERNATE STANDARDS QUANTITATION RELATIONSHIPS

Surrogate/Alternate/Cleanup Standard	Standard Used During Percent Recovery Determination
$^{37}Cl_4$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	13C_{12}-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD

NOTE : For air samples (PUF and M-0023A) recoveries of surrogate standards are quantitated relative to their appropriate internal standards.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

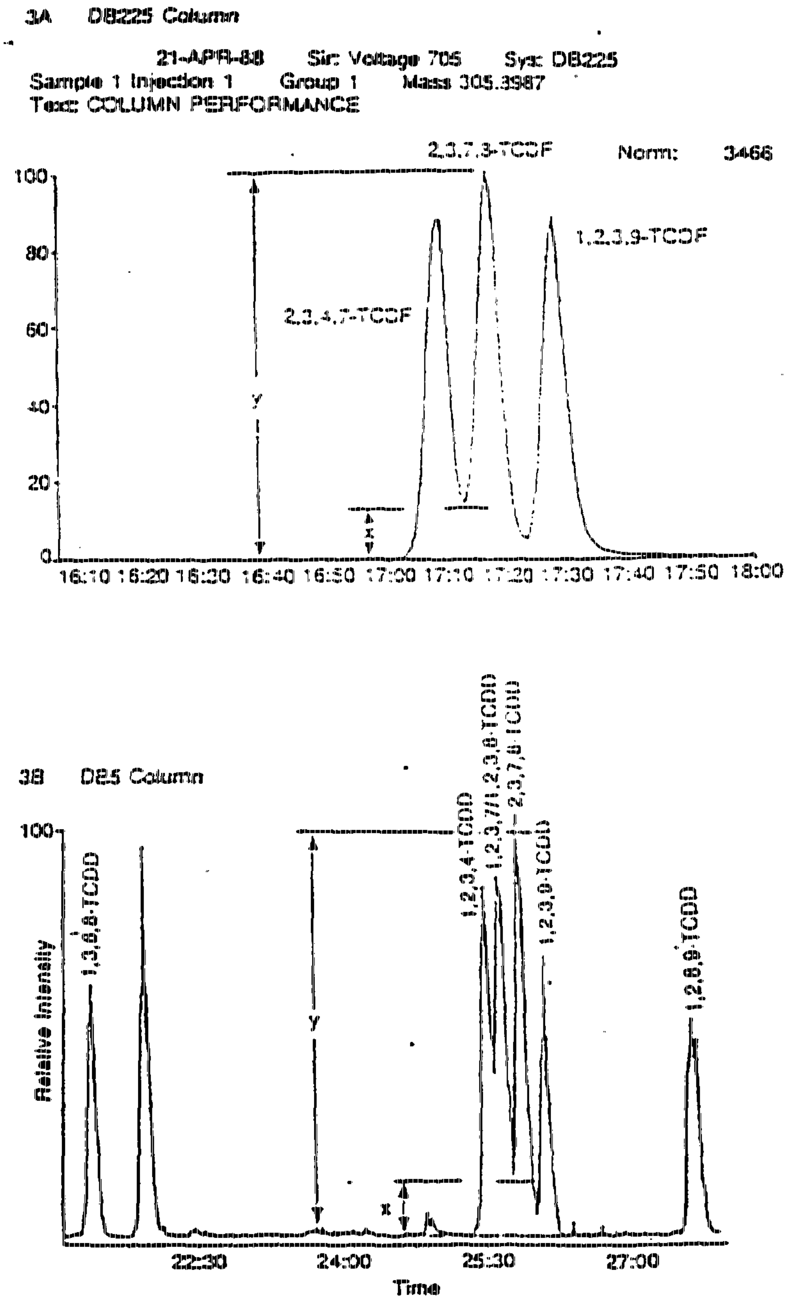

FIGURE 1. Valley between 2,3,7,8 - Tetra Dioxin and Furan Isomers and Other Closely Eluted Isomers.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290	
TLI SOP No DHR182 Version: 6	Date Written: February 9, 1998

APPENDIX 1

Deviations from and improvements to US EPA Method 8290
POLYCHLORINATED DIBENZODIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/ HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/ HRMS)
as performed at Triangle Laboratories, Inc.

See Section

- IV. A. and Table 2. — Triangle Laboratories uses 6 calibration points ranging from 0.5 pg/µL to 200 pg/µL for tetrachlorinated dioxin and furan. Concentrations of penta- through heptachlorinated analytes are 5 times higher than tetra congeners and octachlorinated analytes are 10 times higher (Compare with Table 5., Method 8290).
- IV. A. — Seven more labeled PCDDs/ PCDFs are used as surrogate and alternate standards to provide additional quality control information.
- IV. A. — The 1,2,3,6,7,8 - hexachlorodibenzofuran (HxCDF) is used as an internal standard instead of 1,2,3,4,7,8 - HxCDF. Triangle Laboratories uses 1,2,3,4,7,8 - HxCDF as a surrogate standard.
- IV. A. — Each carbon-labeled standard in the initial calibration solution has a concentration of 100 pg/µL except $^{13}C_{12}$ - OCDD, which is at 200 pg/µL. Concentrations of $^{13}C_{12}$ - labeled standards in sample fortification solutions are the same as in the initial calibration solution (See section 5.9., Method 8290).
- V. — The method blank does not need to be analyzed on each analysis clock that samples are analyzed. Once a valid analysis is provided for the method blank it may be replaced with HRGC/ HRMS solvent blank (See section 8.2., Method 8290).
- V. B. 3. a) — Documentation of the mass spectrometer resolving power is accomplished by recording the peak profile of m/z 416.9760 and 330.9792 (See section 8.2.2.3., Method 8290).
- VII. F. 6. a) — To calculate the sample specific Estimated Detection Limit, noise equivalent area is used instead of noise height (See section 7.9.5.1.1., Method 8290).
- VIII. D. - F. 1. — The Internal Standard recoveries in all QC samples and field samples are considered valid as long as the signal to noise ratio is greater than 10:1, the recovery is $\geq$ 25% and target detection limits for the analytes are met (See section 8.4., Method 8290).
- VIII. E. 2. — For up to two analytes in LCS/ LCSD and MS/ MSD, recoveries may be as high as 145% or as low as 60%, as long as the associated relative percent differences (%RPDs) meet criteria.

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APPENDIX 1 Continued

- VIII. E. 3. For up to two analytes in LCS/ LCSD and MS/ MSD, %RPDs may be higher (up to 35%), as long as the associated percent recoveries meet criteria.

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VOLUME 4C
SITE-SPECIFIC HEALTH AND SAFETY PLAN
for the
ECOLOGICAL RISK ASSESSMENT SUPPORT SAMPLING

SAUGET AREA 2
SITES O, P ,Q, R, S
SAUGET & CAHOKIA, ILLINOIS

Prepared for:

SAUGET AREA 2 SITES GROUP
c/o SOLUTIA, INC.
575 Maryville Centre Drive
St. Louis, MO 63141

Prepared by:

AMEC EARTH AND ENVIRONMENTAL, INC.
285 DAVIDSON AVE.
SOMERSET, NJ 08873

MAY 2001

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ATTACHMENTS

LIST OF ACRONYMS AND ABBREVIATIONS

ABRTF	American Bottoms Regional Water Treatment Facility
BTEX	Benzene, toluene, ethylbenzene, xylenes
CERCLA	Comprehensive Environmental Responsibility Compensation and Liability Act
CPR	Cardiopulmonary resuscitation
DOT	Department of Transportation
HASP	Health and Safety Plan
HSM	Project Health and Safety Manager
IDLH	Immediately Dangerous to Life and Health
IEPA	Illinois Environmental Protection Agency
MSDS	Material Safety and Data Sheet
OSHA	Occupational Safety and Health Administration
PAH	Polynuclear hydrocarbon
PCB	Polychlorinated Biphenyl
PEL	Permissible Exposure Limit
PPE	Personal Protective Equipment
PM	Project Manager
SOPs	Standard Operating Procedures
USCG	United States Coast Guard
USEPA	United States Environmental Protection Agency

1.0 INTRODUCTION

The purpose of this document is to establish standard health and safety procedures for AMEC Earth and Environmental, Inc. (AMEC) employees and their subcontractors in performance of the planned field activities associated with conducting the ecological risk assessment at the Sauget Area 2 Sites O, P, Q, R, and S, located in Sauget and Cahokia, Illinois (Figure 1).

Site activities shall be performed in accordance with AMEC health and safety policies and procedures; applicable Occupational Safety and Health Administration (OSHA) Standards; 29 CFR Part 1910 and 1926; applicable Environmental Protection Agency (EPA) requirements; and consensus standards. Where the word "shall" is used, the provisions of this plan are mandatory.

The levels of personal protection and the procedures specified in this plan are based on the best information available from reference documents and site characterization data. Therefore, these recommendations represent the minimum health and safety requirements to be observed by all personnel engaged in this project. Unforeseeable site conditions or changes in scope-of-work may warrant a reassessment of protection levels and controls stated. All adjustments to the site-specific health and safety plan (HASP) must have prior approval by the AMEC Site Health and Safety Officer (SHSO), the AMEC Corporate Health and Safety Manager (HSM), the AMEC Project Manager, and other designated authorities.

All personnel involved in this project must read this document carefully. Any questions or concerns, which the reader feels are not adequately addressed should be directed to the AMEC SHSO and/or HSM. Personnel on-site shall be required to follow all appropriate health and safety procedures; be alert to the hazards associated with work on a hazardous waste site; be aware of the hazards associated with working over or near water; and exercise reasonable caution at all times. Personnel have the right to refuse to work under conditions which they feel are of a health or safety concern until the concern is appropriately resolved.

All site visitors must receive prior approval from the SHSO and may do so only for the purpose of observing site conditions or operations. Upon arrival, visitors will report to the SHSO where he/she will be logged in the field notebook, required to fill out a medical data sheet and undergo a safety and evacuation orientation (as necessary, given the nature of ongoing work activities).

Visitors will be expected to comply with relevant OSHA requirements such as medical surveillance, training, and respiratory protection as determined by the Site Health and Safety Officer.

2.0 SITE DESCRIPTION AND FEATURES

Sauget Area 2 is located in the City of East St. Louis and the Villages of Sauget and Cahokia in St. Clair County, Illinois. The Sauget Area 2 study area is east of the Mississippi River and south of the MacArthur bridge railroad tracks. The study area is west of Route 3 (Mississippi Avenue) and north of Cargill Road.

Site	Former Use	Municipality
Site O	Sewage Sludge Dewatering	Village of Sauget
Site P	Municipal and Industrial Waste Disposal	City of East St. Louis Village of Sauget
Site Q	Municipal and Industrial Waste Disposal	Village of Sauget Village of Cahokia
Site R	Industrial Waste Disposal	Village of Sauget
Site S	Chemical Reprocessing Waste Disposal	Village of Sauget

These sites are located in an area historically used for heavy industry, including chemical manufacturing, metal refining and power generation, and waste disposal. Currently the area is used for heavy industry, warehousing, bulk storage (coal, refined petroleum, lawn and garden products and grain), waste water treatment, hazardous waste treatment, waste recycling and truck terminals. Four commercial establishments are located at the north end of the study area. No residences are located within the study area. Residential areas closest to Sauget Area 2 are approximately 3,000 feet east of Site P and about 3,000 feet east of Site O. These residential areas are located, respectively, in East St. Louis and Cahokia.

2.1 SITE LOCATION AND PHYSICAL SETTING

Sauget Area 2 is located in the floodplain of the Mississippi River in an area known as American Bottoms. Topographically, the area consists primarily of flat bottom land although local topographic irregularities do occur. Generally, land surface in the American Bottoms slopes from north to south and from east to west toward the Mississippi River. Land surface elevation ranges from 400 to 410 feet above Mean Sea Level (MSL) with little topographic relief.

Sauget Area 2 consists of five former disposal areas, Sites O, P, Q, R and S, adjacent, or in close proximity, to the Mississippi River. These five disposal areas were given letter designations by the

Illinois Environmental Protection Agency (IEPA) in the 1980s. Two of these sites, Sites Q and R, are located on the wet side of the floodwall and levee which is operated and maintained by the US Corps of Engineers and the Metro East Sanitary District. The floodwall is designed to protect the City of East St. Louis and the Villages of Sauget and Cahokia from flooding. Sites O, P and S are located on the dry side of the floodwall and levee.

2.2 PRESENT AND PAST FACILITY OPERATIONS AND DISPOSAL PRACTICES

Each of the five sites in Sauget Area 2 is described below. Maximum chemical concentrations included in these site descriptions were included by USEPA in the AOC and are summarized in Table 1.

2.2.1 Site O

Site O, located on Mobile Avenue in Sauget, Illinois, occupies approximately 20 acres of land to the northeast of the American Bottoms Regional Wastewater Treatment Facility (ABRTF). An access road to the ABRTF runs through the middle of the site. In 1952, the Village of Sauget Waste Water Treatment Plant began operation at this location. In addition to providing treatment for the Village of Sauget, the plant treated effluent from the various Sauget industries.

During its operation the treatment plant received and treated industrial and municipal wastewater. Approximately 10 million gallons per day of wastewater was treated most of which was from area industries.

Four lagoons were constructed at the wastewater treatment plant in 1965 and placed in operation in 1966/1967. Between 1966/67 and approximately 1978, these lagoons were used to dispose of clarifier sludge from the wastewater treatment plant. They were designated as Site O during a site investigation conducted by IEPA in the 1980s. The lagoons were closed in 1980 by stabilizing the sludge with lime and covering it with approximately two feet of clay. Currently, the lagoons are covered with clay and are vegetated.

Parties that USEPA alleges discharged to the Sauget Wastewater Treatment Plant during the time period that the sludge lagoons were in operation included, at a minimum:

- Amax Zinc Corporation,
- American Zinc Company
- Cerro Copper Products Company
- Clayton Chemical Co.
- Darling Fertilizer
- Ethyl Corporation
- Ethyl Petroleum Additives, Inc.
- Midwest Rubber Reclaiming
- Mobil Oil Corporation
- Monsanto Company
- Rogers Cartage Company
- Wiese Planning and Engineering

Parties which own and/or operate, or previously owned and/or operated, portions of Site O include:

- Village of Sauget

The USEPA alleges that soil samples collected from Site O contain VOCs, SVOCs, PCBs, dioxin and metals at concentrations of up to:

VOCs (ppb)

Benzene	30,769
Chlorobenzene	58,974
Ethylbenzene	166,667
4-Methyl-2-Pentanone	7,692
Toluene	29,487
Xylenes	615,385

PCBs (ppb)

Arochor 1232	30,366
Arochor 1242	1,871,795

Dioxin (ppb)

Tetrachlorodibenzo-p-dioxin	170

Metals (ppm)

SVOCs (ppb)

1,2-Dichlorobenzene	606,000
1,3-Dichlorobenzene	112,821
1,4-Dichlorobenzene	1,030,000
1,2,4-Trichlorobenzene	65,300
1,1,1-Trichloroethane	1,410
1,2,4-Trichlorophenol	26,923
Pentachlorophenol	1,620,000
Benzo(a)anthracene	121,795
Chrysene	282,051
Fluoranthene	74,000
Naphthalene	34,615
Phenanthrene	230,000
Pyrene	282,051
2-Methylnaphthalene	160,256
n-Nitrosodiphenylamine	50,000
Butyl Benzyl Phthalate	3,846,154

Cadmium	31
Copper	341
Mercury	6.3
Nickel	136
Zinc	1,398

The USEPA alleges that groundwater samples collected from Site O contain VOCs, SVOCs and metals at concentrations of up to:

VOCs (ppb)	
Benzene	190,000
2-Butanone	62,000
Chlorobenzene	180,000
trans-1,2-Dichloroethene	14,000
Methylene Chloride	52,000
4-Methyl-2-Pentanone	38,000
1,1,2,2-Tetrachoroethane	12,000
Tetrachloroethene	10,000
Toluene	15,000
Trichloroethene	83,000

SVOCs (ppb)	
4-Chloroaniline	780
1,2-Dichlorobenzene	11,000
1,4-Dichlorobenzene	15,000
4-Methylphenol	1,100
Phenol	1,100

Metals (ppb)	
Arsenic	113
Cadmium	11
Lead	6,350

2.2.2 Site P

Site P, which is bounded by the Illinois Central Gulf Railroad tracks, the Terminal Railroad Association tracks and Monsanto Avenue, occupies approximately 20 acres of land located in the City of East St. Louis and the Village of Sauget. It was operated by Sauget and Company as an IEPA-permitted landfill from 1973 to approximately 1984 accepting general wastes, including diatomaceous earth filter cake, from Edwin Cooper (now Ethyl Corporation) and non-chemical wastes from Monsanto. IEPA inspections documented the presence of drums labeled "Monsanto ACL-85, Chlorine Composition," drums labeled phosphorus pentasulfide from Monsanto and Monsanto ACL filter residues and packaging. Site P is currently inactive and partially covered, however, access to the site is not restricted.

Parties which USEPA alleges to have generated, disposed of, released into and/or transported wastes to Site P include:

- Edwin Cooper Petroleum Additive
- Monsanto Chemical Company

USEPA alleges that parties who potentially own, previously owned and/or operated Site P include:

- Chicago Title & Trust Company
- Gulf-Mobile & Ohio Railroad
- Metro East Sanitary District
- Sauget and Company
- Southern Railway System
- Union Electric Company

USEPA alleges that soil samples collected from Site P contain VOCs, SVOCs and metals at concentrations of up to:

VOCs (ppb)	
Toluene	413
Xylenes	450

Metals and Inorgancis (ppm)	
Lead	526
Mercury	3.9
Cyanide	15

SVOCs (ppb)	
1,2-Dichlorobenzene	3,625
1,4-Dichlorobenzene	8,875
Phenol	3,875
Di-n-butyl phthalate	16,250

2.2.3 Site Q

Site Q, a former subsurface and surface disposal area, occupies approximately 90 acres in the Villages of Sauget and Cahokia. This Site is divided by the Alton and Southern Railroad into a northern portion and a southern portion. The northern portion consists of 65 acres bordered on the north by Site R and Monsanto Avenue, The northern portion is bordered on the south by the main track of the Alton and Southern Railroad and property owned by Patgood Inc. On the east, the northern portion of the site is bordered by the Illinois Gulf Central Railroad and the US Army

Corps of Engineers (USACE) flood control levee and on the west the Site is bordered by the Mississippi River.

The southern portion consists of 25 acres, north of Cargill Road and south of the Alton and Southern Railroad. The southern portion is bounded on the west by a ten-foot wide strip of property owned by Union Electric for transmission lines and a spur track of the Alton and Southern Railroad to the Fox Terminal. A barge terminal operated by St. Louis Grain Company is located between the Union Electric property, the spur track and the Mississippi River. Southern Site Q is bordered on the east by the Illinois Central Gulf Railroad and the flood control levee.

Disposal started in the 1950s and continued until the 1970s. Sauget and Company started operation of a landfill south of the River Terminal in 1966 and terminated operations in 1973. This facility took various wastes including municipal waste, septic tank pumpings, drums, organic and inorganic wastes, solvents, pesticides and paint sludges. It also took plant trash from Monsanto, waste from other industrial facilities and demolition debris.

Most of Site Q is covered with highly permeable black cinders. Eagle Marine Industries and Peavy Company, a division of Con-Agra, operate barge terminal facilities in the central part of the northern portion of Site Q. The southern portion of Site Q is used for reclaiming rebar from concrete and for construction debris disposal. A ten-acre site on the northern portion of Site Q is currently used by Rivercity Landscape Supply as a bulk storage terminal for lawn and garden products. Raw landscape products such as mulch, rock and soil are process and packaged are also processed and packed on this portion of the site.

Access to some portions of the site is restricted by fencing and gates. Other parts of the site have unrestricted access.

Site Q is on the west side of the USCOE floodwall. In 1993, during the highest recorded flood in St. Louis' history, Site Q was flooded. USEPA conducted a CERCLA removal action at the northern portion of Site Q in 1995. USEPA conducted a second CERCLA removal action at the southern portion of Site Q beginning in October of 1999 and into early 2000. During this removal action, USEPA excavated over 2000 drums and over 7,000 cubic yards of contaminated soils containing metals, PCBs, and organics. Excavated material was transported by rail to Oklahoma for disposal at Safety Kleen's Lone Elk hazardous waste landfill.

USEPA alleges that the following parties potentially generated, disposed of, released into and/or transported wastes to Site Q;

- AALCO Wrecking Company, Inc.
- Abco Trash Service
- Able Sewer Service
- Ajax Hickman Hauling
- Atlas Service Company
- Banjo Iron Company
- Barry Weinmiller Steel Fabrication
- Becker Iron & Metal Corporation
- Belleville Concrete Cont. Company
- Bi-State Parks Airport
- Bi-State Transit Company
- Boyer Sanitation Service
- Browning-Ferris Industries of St. Louis
- C&E Hauling
- Cargill Inc.
- Century Electric Company
- Circle Packing Company
- Clayton Chemical Company
- Corkery Fuel Company
- Crown Cork & Seal Company, Inc.
- David Hauling
- Dennis Chemical Company, Inc.
- Disposal Service Company
- Dore Wrecking Company
- Dotson Disposal "All" Service
- Dow Chemical
- Edgemont Construction
- Edwin Cooper Inc.
- Eight & Trendy Metal Company
- Evans Brothers
- Finer Metals Company
- Fish Disposal
- Fruin-Colnon Corporation
- Gibson Hauling
- H.C. Fournie Inc.
- H.C. Fournie Plaster
- Hilltop Hauling
- Huffmeier Brothers
- Hunter Packing Company
- Illinois Department of Transportation
- Inmont Corporation
- Lefton Iron & Metal Company
- Mallinckrodt Chemical
- Midwest Sanitation
- Mississippi Valley Control
- Monsanto Company
- Myco-Gloss
- Obear Nestor
- Roy Baur
- Thomas Byrd
- Trash Men Inc.
- United Technologies Corporation
- U.S. Paint Corporation

USEPA alleges that the following parties potentially own, previously owned and/or operated Site Q include:

- Cahokia Trust Properties
- ConAgra, Inc. (lessee)
- Eagle Marine Industries Inc.
- Industrial Salvage & Disposal Company
- Peavey Company
- Phillips Pipe Line Company
- Pillsbury Company (lessee)
- Sauget & Company
- Union Electric Company
- Village of Cahokia
- Village of Sauget

Soil samples collected from Site Q allegedly contain VOCs, SVOCs, metals, PCBs and dioxin. at concentrations of up to:

VOCs (ppb)	
Chlorobenzene	100,000
Ethylbenzene	790,000
4-Methyl-2-Pentanone	250,000
Toluene	2,400,000
Xylenes	2,300,000

PCBs (ppb)	
Aroclor 1248	70,000
Aroclor 1254	360,000
Aroclor 1260	16,000,000

Dioxin (ppb)	
2,3,7,8-TCDD	3.31

SVOCs (ppb)	
1,2-Dichlorobenzene	3,625
1,4-dichlorobenzene	1,200,000
Bis(2-ethylhexyl) phthalate	1,100,000
Di-n-butylphthalate	900,000

Metals (ppm)	
Antimony	17,900
Arsenic	0.216
Cadmium	152,000
Chromium	3,650
Copper	1,630
Lead	195,000
Mercury	4.9
Nickel	371
Selenium	59.5
Silver	30.2
Thallium	0.89
Zinc	9,520

Groundwater samples collected from Site Q contain VOCs, SVOCs, metals and Inorganics at concentrations of up to:

VOCs (ppb)		**SVOCs (ppb)**	
Benzene	2,000	4-Chloroaniline	15,000
Chlorobenzene	6,700	Phenol	190,000
1,2-Dichloroethane	3,000	2-Chlorophenol	33,000
2-Hexanone	3,500	2, 4-Dichlorophenol	14,000
4-methyl-2-pentanone	2,700	2,4,6-Trichlorophenol	6,000
Toluene	1,600	Pentachlorophenol	35,000
		4-Methylphenol	23,000
Metals and Inorganics (ppm)		2,4-Dimethylphenol	2,800
Arsenic	0.100	2-Nitroaniline	2,000
Cyanide	1,560	Acenaphthylene	3,900

2.2.4 Site R

Site R, a closed industrial-waste disposal area owned by Solutia Inc, is located between the flood control levee and the Mississippi River in Sauget, Illinois. Its northern border is Monsanto Avenue and its southern border is Site Q. A portion of Site Q, known as the "Dog Leg", is located to the east of Site R. This site once called the "Sauget Toxic Dump" and the "Monsanto Landfill" it is now known as the "River's Edge Landfill".

Industrial Salvage and Disposal, Inc. (ISD) operated the River's Edge Landfill for Monsanto from 1957 to 1977. Hazardous and non-hazardous bulk liquid and solid chemical wastes and drummed chemical wastes from Monsanto's W.G. Krummrich plant and, to a lesser degree, its Queeny plant in St. Louis were disposed at Site R. Disposal began in the northern portion of the site and expanded southward. Wastes contained phenols, aromatic nitro compounds, aromatic amines, aromatic nitroamines, chlorinated aromatic hydrocarbons, aromatic and aliphatic carboxylic acids and condensation products of these compounds.

In 1979, Monsanto completed the installation of a clay cover on Site R to cover waste, limit infiltration through the landfill, and prevent direct contact with fill material. The cover's thickness ranges from two feet to approximately eight feet. In 1985, Monsanto installed a 2,250-foot long rock revetment along the east bank of the Mississippi River adjacent to Site R. The purpose of the stabilization project was to prevent further erosion of the riverbank and thereby minimize potential for the surficial release of waste material from the landfill. During the 1993 flood, Site R was flooded but the clay cap was not overtopped. No erosion of the river bank or cap resulted from this flood.

Access to Site R is restricted by fencing and is monitored plant personnel.

On February 13, 1992, the State of Illinois and Monsanto signed a consent decree entered in St. Clair County Circuit Court requiring further remedial investigations and feasibility studies to be conducted by Monsanto on Site R. The results of the Remedial Investigation/Feasibility Study were submitted to Illinois EPA in 1994. Solutia made a good faith offer to the IEPA to install an engineered cap and a leachate recovery system in 1997.

Parties who allegedly own, previously owned and/or operated Site R include:

- Cahokia Trust Properties
- Monsanto Company
- Solutia Inc
- Sauget and Company

Sediment samples collected from a drainage ditch around Site R showed VOC concentrations ranging from 0.002 to 0.035 ppm. SVOC concentrations in sediments ranged from 0.045 to 3.99 ppm. PCBs were detected at concentrations ranging from 0.08 to 1.5 ppm. Elevated levels of metals, particularly aluminum, iron and magnesium, were also detected. Sediment samples collected adjacent to the Mississippi River on the west side of Site R showed SVOC concentrations ranging from 0.001 to 7.7 ppm. PCBs were also detected at concentrations ranging from .00001 to .23 ppm.

Soil samples collected from Site R showed elevated levels of VOCs ranging from 0.15 to 5,800 ppm. SVOCs were found at levels ranging from 0.017 to 19,000 ppm. Pesticides were found at levels ranging from 0.011 to 99 ppm and PCBs were detected at levels ranging from 0.075 to 4,800 ppm. Elevated levels of arsenic, chromium, lead, nickel and mercury were also detected in Site R soils.

SVOC concentrations in leachate samples ranged from 0.6 to 12.3 ppb. Pesticide concentrations ranged from 0.5 to 3.0 ppb and PCBs were detected at 0.08 ppb. Dioxin/furan concentrations ranged from 0.0001 to 0.0014 ppm. Cyanide was also detected at 71 ppb.

Groundwater samples collected from wells on and immediately downgradient of Site R had VOCs concentrations of up to 38,136 ppb. SVOCs were detected in concentrations as high as 2,973,885 ppb. Historical groundwater data is presented in the Site Sampling Plan (SSP). The following constituents were detected at the following maximum concentrations:

Upper Hydrogeologic Unit (UHU)

VOCs (ppb)		SVOCs (ppb)	
Acetone	69,000	Aniline	200,000
Benzene	11,300	2-Chloroaniline	300,000
Bromoform	4,700	3-Chloroaniline	100,000
2-Butanone	3,100	4-Chloroaniline	200,000
Chlorobenzene	158,000	2-Nitroaniline	500,000
Chloroethane	10,000	4-Nitroaniline	500,000
Chloroform	1,600		
1,1-Dichloroethane	4,700	1,2-Dichlorobenzene	100,000
1,2-Dichloroethane	16,500	1,3-Dichlorobenzene	100,000
1,1-Dichloroethene	2,800	1,4-Dichlorobenzene	100,000
trans-1,2-Dichloroethene	11,300	1,2,4-Trichlorobenzene	100,000
Methylene Chloride	22,400		
4-methyl-2-pentanone	3,100	Nitrobenzene	100,000
1,1,2,2-Tetrachloroethane	7,000	2-Nitrochlorobenzene	3,400,000
Tetrachloroethene	4,100	3-Nitrochlorobenzene	730,000
Toluene	6,000	4-Nitrochlorobenzene	1,500,000
1,1,1-Trichloroethane	3,800		
Trichloroethene	4,610	Phenol	2,000,000
Vinyl Chloride	24,500	2-Chorophenol	540,000
		4-Chlorophenol	210,000
		2,4-Dichlorophenol	340,000

2,4,6-Trichlorophenol	100,000
3-Methylphenol	280,000
4-Methylphenol	47,000
2,4-Dimethylphenol	100,000
4-chloro-3-Methylphenol	100,000
4-Nitrophenol	500,000
Naphthalene	100,000
2-Chloronaphthalene	100,000
Benzoic Acid	50,800
Benzyl Alcohol	1,830
bis(2-chloroethoxy)methane	100,000
bis(2-ethylhexyl)phthalate	100,000
4-Nitrodiphenylamine	1,250

Middle Hydrogeologic Unit (MHU)

VOCs (ppb)	
Acetone	22,000
Benzene	9,980
Chlorobenzene	60,200
Chloroform	400
Chloromethane	2,500
1,1-Dichloroethane	1,200
1,2-Dichloroethane	9,200
1,1-Dichloroethene	700
trans-1,2-Dichloroethene	11,300
Ethylbenzene	2,500
Methylene Chloride	2,260
4-methyl-2-Pentanone	3,100
Tetrachloroethene	1,050
Toluene	3,000
1,1,1-Trichloroethane	950
Trichloroethene	500

SVOCs (ppb)	
Aniline	685,000
2-Chloroaniline	329,000
3-Chloroaniline	57,200
4-Chloroaniline	105,000
1,2-Dichlorobenzene	25,000
1,3-Dichlorobenzene	25,000
1,4-Dichlorobenzene	25,000
1,2,4-Trichlorobenzene	25,000
Nitrobenzene	25,000
2-Nitrochlorobenzene	463,000
3-Nitrochlorobenzene	460,000
4-Nitrochlorobenzene	185,000
Phenol	1,100,000

Vinyl Chloride	2,500
Xylenes	2,500

2-Chorophenol	160,000
4-Chlorophenol	67,000
2,4-Dichlorophenol	83,000
2,4,6-Trichlorophenol	25,000
Pentachlorophenol	125,000
3-Methylphenol	110,000
2,4-Dimethylphenol	25,000
4-Nitrophenol	125,000
Naphthalene	25,000
Chrysene	25,000
Fluoranthene	25,000
Pyrene	25,000
n-Nitrosodiphenylamine	25,000
bis(2-ethylhexyl)phthalate	25,000

Deep Hydrogeologic Unit (DHU)

VOCs (ppb)	
Acetone	500
Benzene	613
Chlorobenzene	7,380
Chloromethane	500
1,2-Dichloroethane	1,910
trans-1,2-Dichloropropene	720
Ethylbenzene	500
Methylene Chloride	1,790
4-methyl-2-pentanone	1,190
Tetrachloroethene	1,220
Toluene	2,070
Trichloroethene	500
Xylenes	962

SVOCs (ppb)	
Aniline	48,000
2-Chloroaniline	195,000
3-Chloroaniline	52,400
4-Chloroaniline	56,900
2-Nitroaniline	5,000
4-Nitroaniline	5,000
1,2-Dichlorobenzene	9,810
1,3-Dichlorobenzene	950
1,4-Dichlorobenzene	2,250
1,2,4-Trichlorobenzene	950
Nitrobenzene	1,010
2-Nitrochlorobenzene	219,000
3-Nitrochlorobenzene	30,900

4-Nitrochlorobenzene	115,000
Phenol	33,000
2-Chorophenol	8,500
4-Chlorophenol	18,000
2,4-Dichlorophenol	12,800
2,4,6-Trichlorophenol	3,030
Pentachlorophenol	2,500
2,4-Dimethylphenol	1,400
Benzo(a)pyrene	1,300
Benzo(k)fluoranthene	1,300
Chrysene	1,300
Fluoranthene	1,100
Naphthalene	800
Pyrene	950
4-Nitrodiphenylamine	5,000
n-Nitrosodiphenylamine	1,900
bis(2-chloroethyl)Ether	2,900
bis(2-cloroisopropyl)Ether	2,900
bis(2-ethylhexyl)Phthalate	5,000
di-n-butylphthalate	5,000
3,3'-Dichlorobenzidene	8,500
Hexachlorocyclopentadiene	10,000

2.2.5 Site S

Site S is located southwest of Site O. Allegedly, the property is or was owned by the Village of Sauget and the Resource Recovery Group. In the mid-1960s, solvent recovery began on site under Clayton Chemical, which is now owned by the Resource Recovery Group (RRG). The waste solvents were steam-stripped resulting in still bottoms that were allegedly disposed of in a shallow, on-site excavation that is now designated Site S. Historical aerial photographs indicate that Site S was potentially a waste and/or drum disposal area. The northern portion of the site is grassed and its southern portion is covered with gravel and fenced.

Soil samples collected from Site S allegedly contain VOCs, SVOCs, PCBs and metals at concentrations up to:

VOCs (ppb)		**SVOCs (ppb)**	
Ethylbenzene	450,000	Naphthalene	200,000
4-Methyl-2-Pentanone	93,000		
Toluene	990,000	Bis(2-ethylhexyl)phthalate	20,000,000
1,1,1-Trichloroethane	12,000	Butyl Benzyl Phthalate	490,000
Xylenes	620,000	Di-n-butyl phthalate	1,500,000
		Di-n-octyl phthalate	310,000
PCBs (ppb)		**Metals (ppm)**	
Aroclor 1248	85,000	Copper	139
Aroclor 1254	69,000	Lead	0.392
Aroclor 1260	41,000	Mercury	3.5
		Zinc	327

Additional information regarding environmental investigation at the Sites may be found in the Site Sampling Plan.

3.0 SITE ORGANIZATION AND COORDINATION

AMEC will have overall responsibility for the conduct of the activities associated with the ecological risk assessment conducted as part of the overall remedial investigation at the Sauget Area 2 sites, including management of the performance of the necessary subcontractors. The following section details the organizational structure for this project. Key personnel and their project responsibilities are listed in Table 2, Project Organization and Roles.

3.1 SITE HEALTH AND SAFETY OFFICER (SHSO) ROLE

The SHSO reports to the Project Manager and to the HSM for all aspects of the project and is the primary on-site contact for health and safety during field activities. The SHSO oversees the on-site execution of all field activities regarding health and safety procedures and subcontractor operations. The SHSO has the authority to stop all work if conditions are judged to be hazardous to on-site personnel or to the public. The SHSO also has the authority to temporarily suspend workers from the job site for serious violations of the HASP. Other specific responsibilities are as follows:

1. Require specific health control precautions prior to work area entry by AMEC personnel, subcontract personnel, or visitors including briefing of personnel on project and potential hazards, ensuring personnel have reviewed the HASP, on-site safety meetings, and reviewing planned emergency response procedures.

2. Require any AMEC or subcontract personnel to obtain immediate medical attention in the case of a work-related injury or illness.

3. May order work to cease and require evacuation of the work area by all personnel. Has the power to re-establish safe working conditions.

4. Control access to the site by visitors. Advise visitors of their responsibility before entry is allowed.

5. Ensure the correct field execution of the HASP including workplace and personnel monitoring.

6. Perform monitoring/sampling of site hazards for exposure and hazard evaluation.

7. Advise emergency response personnel in an emergency.

8. Coordinate and minimize the number of personnel and amount of equipment in the exclusion zone required for safe and effective site operations.

9. Calibrate all monitoring equipment that will be used on a daily basis and record the results in the daily calibration log.

10. Coordinate accident prevention plan by oversight of field activities and being aware of site operations.

11. The SHSO has the primary responsibility for investigation of injuries, illnesses, and "near misses", and completion of appropriate documentation.

3.2 Project Health and Safety Manager (HSM) Role

The HSM is responsible for coordinating development and approval of HASP and coordinating the execution of health and safety procedures. The HSM assists and advises on resolution of health and safety issues when needed. The HSM, as necessary given the complexity of site work or potential risk associated with investigation of site materials, is responsible for on-site briefing of HASP and support staff prior to commencement of work.

3.3 Project Manager Role

The Project Manager is the primary on-site contact for the Sauget Area 2 project. The Project Manager will coordinate access and security to the site and will advise AMEC personnel, subcontractors, and visitors of proper access and security procedures. The Project Manager is the primary contact for changes in scope of work and coordinating efforts between AMEC and Solutia relative to ecological sampling activities at the Sauget Area 2 sites. The Project Manager will issue the Notice to Proceed with on-site investigation after approval of work plan and HASP.

3.4 FIELD TEAM LEADER

The Field Team Leader is responsible for management of AMEC field effort and oversees the field efforts of AMEC personnel and subcontractors, and provides relevant technical and project performance information to the AMEC Project Manager. The Field Team Leader is responsible for coordination with the site-health and safety officer for implementation of the HASP.

3.5 FIELD PERSONNEL ROLE

AMEC and subcontract personnel who will be involved in the on-site execution of supervision, monitoring, testing, or sampling activities are responsible for:

1. Taking all reasonable precautions to prevent injury to themselves and to their fellow employees; being alert to potentially harmful situations.
2. Performing only those tasks that they believe they can do safely, and immediately reporting any accidents and/or unsafe conditions to the SHSO and/or the Site Manager.
3. Notifying the SHSO of any special medical conditions (*e.g.*, allergies, contact lenses, pregnancy, diabetes) and, if necessary, ensuring that all on-site personnel are aware of the condition.
4. Notifying the SHSO of any prescription and/or non-prescription medication, which the worker may be taking that might cause drowsiness, anxiety or other unfavorable side affects, or may interfere with administration of medical treatment in the unlikely occurrence of an emergency or potential overexposure.
5. Preventing spillage to the extent possible. In the event spillage occurs, contain the spillage, notify the SHSO, and clean up immediately using safe clean up measures as directed by the SHSO. Do not engage in spill containment or clean up if conditions are not safe.
6. Avoid splashing materials to the extent possible.
7. Practicing good housekeeping by keeping the work area neat, clean, and orderly to the extent possible.
8. Reporting all injuries and "near misses," no matter how minor.

9. Complying with the HASP and all health and safety recommendations, precautions, and proper use of the different levels of personal protective equipment as determined by the HASP and/or the SHSO.

4.0 SCOPE OF WORK/PLANNED SITE ACTIVITIES

4.1 SAMPLING OBJECTIVES

The objective of the sampling program is to collect sufficient information to determine if contaminants from the Sauget Area 2 sites have adversely impacted either the terrestrial or the aquatic ecosystems.

4.2 SAMPLING PROCEDURES

Aquatic and terrestrial sampling will be conducted in accordance with the procedures outlined in the Sauget Area 2 Site Sampling Plan.

4.2.1 Aquatic Ecological Activities

The following sections summarize the sampling activities associated with sample collection for the aquatic ecosystem portion of the risk assessment.

4.2.1.1 Surface Water Samples

Sixty surface water samples will be collected from the Mississippi River. Sampling will be conducted from a boat; thus, activities will be performed in accordance with OSHA requirements for "working over or near water" (29 CFR 1926.106) as described in Section 6.3.12 of this HASP in addition to OSHA HAZWOPER procedures (29 CFR 1910.120).

4.2.1.2 Sediment Sampling

Sixty sediment samples will be collected from the surface water locations within the Mississippi River; thus, activities will be performed in accordance with OSHA requirements for "working over or near water" (29 CFR 1926.106) as described in Section 6.3.12 of this HASP in addition to OSHA HAZWOPER procedures (29 CFR 1910.120).

4.2.1.3 Biota Sampling

Sixty fish samples will be collected from the surface water locations within the Mississippi River; thus, activities will be performed in accordance with OSHA requirements for "working over or near water" (29 CFR 1926.106) as described in Section 6.3.12 of this HASP in addition to OSHA HAZWOPER procedures (29 CFR 1910.120).

4.2.2 Terrestrial Ecological Activities

The following sections summarize the sampling activities associated with sample collection for the terrestrial ecosystem portion of the risk assessment.

4.2.2.1 Vegetation Sampling

A total of 24 samples of vegetation will be collected, by hand using stainless steel scissors or shears, at the location of soil/waste characterization samples that will be collected as part of the remedial investigation.

4.2.2.1 Macroinvertebrate Sampling

A total of 24 earthworm samples will be taken by digging with a decontaminated hand trowel or shovel, at soil/waste characterization sample locations collected as part of the remedial investigation.

5.0 WASTE CHARACTERIZATION

WASTE TYPES: (Check all that apply)

(X) Liquid (X) Sludge ( ) Unknown

(X) Solid ( ) Gas

WASTE CHARACTERISTICS: (Check all that apply)

(X) Corrosive (X) Flammable ( ) Radioactive

(X) Toxic (X) Volatile (X) Reactive

( ) Inert (X) Carcinogenic ( ) Unknown

HAZARDOUS MATERIALS SUMMARY: (Check all that apply)

Chemicals

( ) Acids (X) Metals (X) Phenols

( ) Caustics (X) Pesticides ( ) Paints

(X) Halogen (X) PCBs (X) Solvents

(X) Other: PAHs, anilines, dioxins, phthalates

Oils/Fuels

( ) Fuel oil ( ) Gasoline ( ) Diesel

( ) Other:

Sludges

( ) Metal sludges ( ) Oily sludges (X) Septic sludges

(X) Other: diatomaceous earth filter cake

Solids

( ) Asbestos (X) Landfill refuse ( ) Tailings

( ) Other:

6.0 HAZARD EVALUATION

Chemical, physical and biological hazards occur at the site. This section addresses these hazards as well as hazards specific to work tasks.

6.1 HAZARD ANALYSIS OF WORK TASKS

Surface Water/Sediment/Biota Collection

Potential Hazards: (Check all that apply to either existing conditions or are a result of site operations)

(X) Moving Machinery
( ) Projectiles
( ) Confined Space

() Heat Stress
(X) Physical Exertion
(X) Biological

(X) Cold Stress
( ) Noise (>85 dBA)
( ) Electrical (utilities)

(X) Heavy Equipment
(X) Vehicle Traffic
(X) Chemical Exposure

( ) Intrusive Activity
( ) Fire/Explosion
(X) Slips, trips and falls

(X) Other: Working Over or Near Water

Control or Protective Measures: (Check all that apply)

(X) Tailgate Meetings
(X) PPE
(X) Safe Work Practices

(X) Operator Training
( ) Site Control
(X) Decontamination

(X) Engineering Controls
(X) Other: USCG-approved life preserver/ring buoy; fire extinguishers

INITIAL LEVEL OF PERSONAL PROTECTIVE EQUIPMENT (PPE) FOR ASSIGNED TASK:

Initial levels of PPE have been assigned for this work task per the potential for exposure. Levels may be upgraded or downgraded depending on monitoring data and site conditions, as determined by the protocol outlined in Section 11.0 Exposure Monitoring and deemed necessary or appropriate by the SHSO.

Level of protection:	( ) A	( ) C	() Modified D
	( ) B	(X) D	
Respirator:	( ) SCBA, Airline	( ) Fullface Resp	( ) 1/2 Face Resp.
(Level C or above)	( ) OV/HEPA Combo Cart.		( ) Other Cart.:
Protective clothing:	( ) Encapsulating Suit	( ) Tyvek	( ) PE Tyvek
	( ) Saranex	(X) Splash Suit	( ) Other
Head/eye/ear:	( ) Hard Hat	( ) Safety Glasses	( ) Goggles
	( ) Splash Shield	( ) Ear Plugs	( ) Other
Gloves: (Outer/Inner)	(X) Nitrile (outer)	( ) Neoprene	( ) PVC
	(X) Latex (inner)	( ) Vinyl	( ) Other: Leather
Footwear:	( ) Safety-toed Leather		( ) Chemical Overboots
	(X) Safety-toed Rubber		() Other:

Terrestrial Vegetation/Earthworm Collection

Potential Hazards: (Check all that apply to either existing conditions or are a result of site operations)

( ) Rotating Machinery	( ) Projectiles	( ) Confined Space
( ) Heat Stress	(X) Physical Exertion	(X) Biological
(X) Cold Stress	( ) Noise (>85 dBA)	( ) Electrical (utilities)
( ) Heavy Equipment	( ) Vehicle Traffic	(X) Chemical Exposure
(X) Intrusive Activity	( ) Fire/Explosion	(X) Slips, trips and falls
( ) Other:		

Control or Protective Measures: (Check all that apply)

(X) Tailgate Meetings	(X) PPE	(X) Safe Work Practices
(X) Operator Training	(X) Site Control	(X) Decontamination
( ) Engineering Controls	( ) Other:	

INITIAL LEVEL OF PERSONAL PROTECTIVE EQUIPMENT FOR ASSIGNED TASK:

Initial levels of PPE have been assigned for this work task per the potential for exposure. Levels may be upgraded or downgraded depending on monitoring data and site conditions, as determined by the protocol outlined in Section 11.0 Exposure Monitoring and deemed necessary or appropriate by the SHSO.

Level of protection:	( ) A	(X) C	( ) Modified D
	( ) B	( ) D	
Respirator:	( ) SCBA, Airline	(X) Fullface Resp	( ) 1/2 Face Resp.
(Level C or above)	(X) OV/HEPA Combo Cart.		( ) Other Cart.:

Protective clothing: ( ) Encapsulating Suit (X) Tyvek ( ) PE Tyvek

( ) Saranex ( ) Splash Suit ( ) Other

Head/eye/ear: (X) Hard Hat (X) Safety Glasses ( ) Goggles

( ) Splash Shield ( ) Ear Plugs ( ) Other

Gloves: (Outer/Inner) (X) Nitrile (outer) ( ) Neoprene ( ) PVC

(X) Latex (inner) ( ) Vinyl ( ) Other: Leather

Footwear: (X) Safety-toed Leather (X) Chemical Overboots

( ) Safety-toed Rubber ( ) Other:

The hazard analysis for the aquatic sampling (including surface water, sediment, and biota sample collection) and terrestrial sampling (including vegetation and macroinvertebrate) is summarized in Table 3.

6.2 CHEMICAL HAZARDS

The list of chemicals of concern was previously presented as Table 1 and includes the maximum constituent concentrations in soil from previous studies in Sauget Area 2. Potential relevant exposure routes, permissible exposure limits (PELs) and toxicological data for chemicals detected at Sites O, P, Q, R, and S are presented in Table 4.

6.2.1 Aquatic Sampling Activities

The primary route of exposure during surface water and sediment sampling is dermal exposure; however, based on the large volume of water and sediment flow within the Mississippi River, contaminant concentrations are not expected to be a health concern. However, as a precaution, PPE requirements will be strictly enforced and personnel and sampling equipment will be properly decontaminated to eliminate any potential for incidental exposure. Any identified changes in chemical hazards from the original site characterization will necessitate the reassessment of this plan.

6.2.2 Terrestrial Sampling Activities

Prior investigatory work performed at the sites indicate contamination of soil with PCBs, metals, dioxins, and other organic compounds (e.g., chlorobenzene, anilines, chlorophenols, BTEX[1], PAHs, phthalates, chlorinated hydrocarbons). Therefore, potential routes of exposure for the collection of terrestrial biota samples are dermal contact and inhalation exposure to contaminated soils.

6.3 PHYSICAL HAZARDS

6.3.1 Heat Stress

Heat stress monitoring of workers and the environment will be initiated by the SHSO when the ambient temperature exceeds 70°F and workers are dressed out in modified Level "D" protective clothing or greater. To prevent heat stress, personnel monitoring (checking pulse rate or body temperature) will be used as well as scheduled work/rest periods. Workers determined by the SHSO to be displaying symptoms of advanced heat stress will be promptly referred to the designated local hospital.

Work/rest periods will be adjusted based on results of personnel monitoring. The warning symptoms of heat stress include fatigue; loss of strength; reduced accuracy; comprehension and retention; and reduced alertness and mental capacity. Heat stroke represents an advanced form of heat stress and is associated with physical symptoms of hot, dry skin, elevated body temperature (>104 °F), rapid pulse rate, and advanced symptoms of dizziness, nausea, and confusion which may lead to delirium, convulsions, coma, and possible death.

Personnel shall monitor themselves for heat stress as instructed by the SHSO. Self-monitoring shall include checking of pulse rate within two to five minutes into the work break. A pulse rate >110 beats per minute (bpm) shall require shortening of the next work period by 1/3 the time. A pulse rate recorded >110 bpm upon the next rest period shall warrant the same action, and so on. The taking of oral temperatures is not planned at this time, but may be enacted when pulse rate

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[1] benzene, toluene, ethylbenzene, xylenes

measurements exceed 110 bpm. An oral temperature taken during this time period which is >99.6°F shall warrant shortening the next work period by 1/3 the time. A worker recording an oral temperature >100.6°F shall be prohibited from working until their temperature returns to 98.6°F. Heat stress monitoring will be coordinated and documented by the SHSO.

6.3.2 Cold Stress

Cold stress results from the effects of low ambient temperatures and wind velocity. Windchill is the cooling effect wind has on exposed skin. Wind velocity can be estimated using the following guidelines:

- 5 mph: light flag moves
- 10 mph: light flag fully extended
- 15 mph: raises newspaper sheet
- 20 mph: blowing/drifting snow

A windchill chart is provided as Table 5.

Three types of cold stress exist. The first is hypothermia, a lowering of the body's core temperature. It is caused by low (not necessarily freezing) temperatures and is aggravated by hunger, wetness, tiredness, and overexertion. Symptoms include shivering and abnormal behavior such as decreased efficiency, decreased level of communication, forgetfulness, repetitive behavior, poor motor skills, poor judgement, and lack of concern for one's usual physical needs. Prolonged hypothermia results in listlessness, sleepiness, weakness, an inability to walk resulting in repeated falling, stupor, unconsciousness, and ultimately, death.

Treatment of hypothermia is to prevent further heat loss. Victims should be moved to warm, dry areas out of the wind, cold, snow or rain. Wet or damp clothing should be removed and replaced with dry clothing. Cover the person's head as over 60% of body heat is lost through your head. Administer hot fluids if the victim is conscious and monitor the victim's temperature every 15 minutes to ensure proper body temperature (98.6°F).

Frost nip is a local cold injury where the skin is whitened and the victim feels a burning sensation. Move the person to a warm environment and warm the affected area.

Frost bite is caused by exposure to low temperatures and affects the extremities most often. The skin will appear cold, hard, and white. Blisters may form. The victim will have no pain sensation and may have mental confusion and impaired judgement. Ultimately, the person will die.

The aquatic and terrestrial sampling is schedule for October 2001; therefore, weather conditions prevalent during this time of year are conducive to producing cold stress. Cold stress monitoring of workers and the environment will be initiated by the SHSO when the ambient temperature is below 50°F. To prevent cold stress, personnel monitoring will be used as well as scheduled work/rest periods (Table 6). Workers determined by the SHSO to be displaying symptoms of advanced cold stress will be promptly referred to the designated local hospital.

6.3.3 Confined Space

Confined spaces will not be encountered during the ecological sampling activities.

6.3.4 Slip, Trip, And Fall Hazards

Slip, trip, and fall hazards will be minimized by the following housekeeping practices at all work sites:

- Loose or light material and debris will be stored in designated areas and/or containers.
- Tools, materials, extension cords, hoses, or debris will be located so as not to cause tripping or other hazards.
- Tools, materials, and equipment subject to displacement or falling will be adequately secured.
- All efforts should be made to work on level ground. Work areas may have to be altered to ensure a safe, reasonably level area. The sites will be examined before work commences to select the safest areas to set up.

- Any incidental spillage of fuel or fuel oil will be cleaned immediately with absorbent material, and covered with sand or soil to preclude slippage.

- Rubber-soled shoes will be required for those personnel working on the boat.

6.3.5 Noise

Noise levels greater than 85 dBA are not anticipated at the Sauget Area 2 sites.

6.3.6 Lifting

- Workers will use proper lifting techniques, lifting with the legs and not the back. Loads >50 lbs. require a second person or mechanical device.

- Whenever possible, mechanical devices such as drum dollies or hand trucks should be used to lift or move heavy loads.

6.3.7 Electrical Hazards

Electrical hazards may be encountered aboard the boat during sediment sampling for the ecological risk assessment. The boat is equipped with a fire extinguisher and motor kill switch.

6.3.8 Machinery Hazards

Machinery hazards may be encountered during sediment sampling when operating the dredge. The dredge will be operated only by persons qualified by training or experience to operate such equipment or machinery in accordance with OSHA regulations (29 CFR 1926.20).

6.3.9 Engulfment

Engulfment hazards will not be encountered during sampling for the ecological risk assessment.

6.3.10 Nuisance Dusts

Nuisance dusts are not an issue at these sites. Personnel collecting samples for the terrestrial aspect will be required to conduct sampling activities with Level C protection due to the presence of chemical contaminants in soil.

6.3.11 Fire/Explosion

The boat is equipped with a fire extinguisher and motor kill switch. Smoking will not be allowed on the boat during engine operation. Complete an Incident Report (Attachment B) within 24 hrs. for all work shutdowns.

6.3.12 Inclement Weather

Work shutdown conditions are as follows:

- Poor visibility.
- Precipitation severe enough to impair safe movement or travel.
- Lightning in the immediate area.
- Terrestrial sampling: steady winds greater than 40 mph. Aquatic sampling: winds greater than 20 mph.
- Other conditions as determined by the SHSO, Field Team Leader or HSM.
- Complete an Incident Report (Appendix B) within 24 hrs. for all work shutdowns.

6.3.13 Working Over or Near Water

In accordance with OSHA regulations for working over or near water (29 CFR 1926.106) and Coast Guard boating safety regulation, persons shall wear a US Coast Guard-approved life jacket or buoyant work vest at all times while working on the boat. Prior to and after each use, the vests or life preservers shall be inspected for defects which would alter their strength or buoyancy. Defective units shall not be used.

The boat to be used during the aquatic sampling shall be equipped with proper lights, air horn, first aid kit, fire extinguisher, cell phone, railings on three sides, motor kill switch, full spare gasoline tank, oars, installed oar locks, and an anchor. Coolers packed with liquid replenishment and food are allowed on board; however, no eating or drinking will occur during the handling/collection of hazardous materials or biota samples before personnel thoroughly was their hands.

The boat captain will be assigned prior to boating activities and all personnel working on board shall follow his/her orders. The boat captain shall be responsible for knowing and following US Coast Guard guidelines relative to boat traffic and/or other boating safety issues (www.uscgboating.org/reg/regfrcontents.asp). No smoking is allowed at any time in the boat or within 50 feet of it during refueling. All personnel on board shall be responsible for being cautious of coiled and frayed lines or lines under tension while in or near the boat. In addition, all personnel shall carefully lower lines into the water and never let a weighted line free fall. The depth to the bottom of the River shall be known before deploying equipment. Personnel shall remain seated in the boat unless deploying equipment. In this case, they shall keep their center of gravity as low as possible and as near as possible to the center of the boat. Refer to the control measures listed in Table 3 (Activity Hazard Analysis) for boating operations.

For persons working on shore, ring buoys with at least 90 feet of line shall be provided and readily available for emergency rescue operations. Distances between ring buoys shall not exceed 200 feet. At least one lifesaving skiff shall be immediately available at locations where employees are working over or adjacent to water.

6.4 BIOLOGICAL HAZARDS

Biological hazards at the Sauget sites include potential exposure to poison ivy, tetanus, mosquitoes, rodents, and ticks.

To reduce the chances of dermatitis caused by poison ivy, personnel should wear long sleeves and long pants. Any exposed skin should be wiped downed several times a day with baby wipes or apply a blocking product (e.g., "Ivy Block"). Long sleeves and pants will also help to prevent mosquito bites.

Many of the study areas are former landfills and may have protruding objects. Therefore, field personnel will be required to have a current tetanus shot prior to job initiation.

Contact with rodents should be avoided during field work.

Ticks are vectors for a number of diseases such as Rocky Mountain spotted fever or Lymes disease. Lymes disease can result in permanent damage to the nervous system and joints. Ticks are found throughout the United States.

Ticks are tiny and are found in brush, woods, and tall grass. May and June are the worst months for potential exposure, but the ticks are active in all months when the temperature exceeds 45°F. Preventative measures to avoid exposure to ticks include the following:

- Field personnel should wear long pants and long-sleeved shirts. It is recommended that pant cuffs be placed inside the work boots. Shirts should be tucked in. The use of light colored clothing may make it easier to spot ticks.

- Tick repellent should be applied to clothing and skin. Permethrin-containing repellent may be sprayed near the openings on work clothes (pant bottoms and waistband); permethrins should not be used on skin. On skin, DEET (N-diethyl-metatoluamide)-containing repellent (< 33% DEET) should be applied. DEET should not be used on the face.

- All personnel should check for the presence of ticks or tick bites every day. Skin and hair should be checked for ticks. Many people get a spot on their skin in three to 30 days following a tick bite; the spot looks like a small red bullseye that is spreading out.

- If a tick is observed on the skin, it should be removed immediately: hold a tweezer on the tick as close to the skin as possible, and pull. Kill the tick and retain for examination by a physician. Performance of a blood test is appropriate following exposure to a tick bite. The Lyme blood test may not indicate positive results until two weeks or more after a tick bite. (Note: Ticks should not be killed with or stored in any liquid as this makes testing for Lymes disease impossible).

7.0 HAZARD COMMUNICATION AND TRAINING ASSIGNMENTS

Potential hazards shall be communicated during the sample handling process. Samples shall be identified as to their potential hazard and packaged to prevent spillage or breakage in accordance with Department of Transportation (DOT) regulations. Any unusual sample conditions shall be noted. In addition, the laboratory receiving the samples shall be advised of the potential contaminants present and associated hazard level (either through phone conversation or documentation with the samples).

Hazards associated with the chemicals used during the sample preservation and equipment decontamination (including hydrochloric acid, nitric acid, and methanol) shall be communicated to the field personnel.

7.1 HAZARD COMMUNICATION TRAINING

All site personnel shall be trained in the hazards associated with this project prior to commencement of work activities. Material safety data sheets (MSDSs) for all materials on-site shall be kept on-site and available to all workers at all times (refer to Attachment C). AMEC shall be responsible for acquiring all pertinent MSDSs and the SHSO shall be responsible for upkeep of the MSDS file at the job site.

7.2 HAZARDOUS WASTE WORKER TRAINING

All site staff will have completed the OSHA 40-hour Hazardous Waste Operations Training, 24-hour on-site supervised training, and appropriate annual updates [29 CFR 1910.120(e)]. In addition, Site Supervisors will have completed OSHA 8-hr Supervisory Training. First Aid and cardiopulmonary resuscitation (CPR) trained personnel shall have Bloodborne Pathogen Training as mandated by OSHA 29 CFR 1910.1030. Occasional site workers that will not receive exposures exceeding permissible exposure limits require, at a minimum, 24 hours of OSHA Hazardous Waste Operations training and one day on-site training and supervision. Documentation that training assignments have been met will be required prior to site entry for AMEC personnel and their subcontractors.

7.3 First Aid/CPR Training

A minimum of one on-site person will be trained in basic first aid and CPR as administered by American Red Cross or National Safety Council. A first aid kit will be available at all times that work is in progress and will be stored in a field vehicle and the boat. All on-site personnel will be advised of its location.

7.4 Orientation Training And Daily Safety Meetings

An on-site orientation session will be required for all site personnel, including subcontractors and visitors, prior to commencement of work and will include the following:

- Overview of planned activities, procedures and monitoring techniques
- Health effects and hazards of the chemicals identified or suspected to be on-site
- Personnel protection, including:
  - Use, care and fitting of PPE
  - Necessity for personnel protection, its effectiveness and limitations
- Decontamination procedures
- Prohibitions in work areas
  - Site layout
  - Standard safe work practices
- Emergency procedures, including:
  - Emergency contacts
  - Instructions for implementing the emergency plan
  - Map of site layout

A daily safety meeting will be conducted and documented at the beginning of each workday or work shift. Health and safety considerations for the day's activities will be discussed and protective equipment necessary will be outlined. Problems related to personal protection, inclement weather, or the interpretation of newly available environmental monitoring data will be topics typically covered during these briefings. These meetings will be documented in the field notebook.

8.0 MEDICAL SURVEILLANCE REQUIREMENTS

All AMEC personnel and their subcontractors working on-site shall participate in a medical surveillance program which is consistent with the requirements of 29 CFR 1910.120 for hazardous waste site operations. The purpose of the program is to assess and monitor employee health prior to employment, during the course of, and at the termination of employment. Subcontractor employees are required to have been certified by their employer that they are in compliance with this requirement and provide proof to AMEC prior to job initiation.

8.1 COMPREHENSIVE PHYSICAL EXAM

The program consists of scheduled baseline exams, follow-ups, termination exams, and other exams as needed. The basic exam protocol is as follows:

1. An occupational and medical history;
2. A physical exam;
3. Whole body count;
4. Laboratory tests including blood chemistry, CBC, reticulocyte count (heavy metals specific for chromium, lead and mercury, and chlorinated hydrocarbons specific for PCBs);
5. Urinalysis;
6. EKG;
7. Stool guaiac;
8. Spirometry with chest x-ray;
9. Audiometric testing;
10. Vision tests; and
11. Respirator quantitative fit test.

Copies of medical clearances, training records, and respirator qualification cards shall be made available to, and copies kept by, the SHSO prior to each individual on-site worker beginning site duties. Medical records and exposure records clearance for each on-site worker will be maintained in their individual personnel file at their respective office of employment.

8.2 EMERGENCY MEDICAL TREATMENT

Personnel who exhibit signs and symptoms of chemical or heat overexposure, or have been injured on the job, might also seek medical services. See also the Emergency Response (Section 17) for specific information regarding emergency services and logs, reports, and record keeping. Subcontractors should provide internal Workers' Compensation information to the SHSO or Project Health and Safety Manager during the pre-work meeting, for emergency use.

9.0 COMMUNICATIONS

The "buddy system" will be enforced for field activities involving potential exposure to hazardous or toxic materials. Each person will observe their partner for symptoms of chemical overexposure or cold/heat stress and provide emergency assistance when warranted. An audible emergency signaling device shall be maintained in the field.

The following emergency signals shall be used:

- Thumbs up — OK; understand
- Thumbs down — No; negative
- Grasping buddy's wrist — Leave site now
- Hands on top of head — Need assistance
- (Air Horn) One long blast — Evacuate site
- (Air Horn) Two short blasts — Return to site

10.0 SANITATION/ILLUMINATION

Washing facilities (potable water, soap, and disposable towels) shall be available on-site for use by field personnel. Local restroom facilities will be identified by the Site Superintendent/SHSO for use by project personnel. Work will only be conducted during daylight hours.

11.0 EXPOSURE MONITORING

Available site information does not warrant the anticipation of Immediately Dangerous to Life and Health (IDLH) concentrations. The levels of protection outlined in Section 6.1 (Hazard Analysis of Workers Tasks) have been assigned in accordance with known concentrations of materials. Level D has been assigned for aquatic sampling due to the potential for dermal contact with contaminated sediments. Level C has been initially assigned for terrestrial sampling due to the potential for inhalation and dermal contact with contaminated soils.

11.1 TYPE AND FREQUENCY OF AIR MONITORING

Some of the compounds detected in soil exhibit vaporous qualities; therefore, air monitoring will be conducted according to Table 3.

11.2 MONITORING INSTRUMENTS

The SHSO will maintain equipment SOPs (Attachment D) onsite that specify calibration, general use, and troubleshooting procedures. The photoionization detector will be field calibrated on a daily basis according to the manufacturers instructions, and will be recorded on the calibration log (Attachment D).

11.3 PERSONAL SAMPLING

It is not necessary to selectively monitor site workers for specific parameters with laboratory confirmation for this project.

11.4 ACTION LEVELS

Action levels shall be established for upgrading/downgrading PPE, work stoppages, and evacuation. The decision to upgrade/downgrade the level of PPE must be based upon instrument readings measured in the breathing zone of site personnel and comparison of the results to the information contained in Table 7. Readings shall be recorded in the field notebook.

12.0 WORK ZONE DELINEATION

Based on site characterization information previously obtained from the project area, hazardous materials are expected to be encountered during proposed data collection activities. A centralized area at each site (i.e., O, P, Q, R, S) will be established for equipment decontamination and disposal of used PPE. The establishment of working zones is described below.

12.1 EXCLUSION ZONE

The exclusion zone is the area where the potential for worker exposure with contaminated materials is greatest. All personnel entering this zone will be required to have the prescribed level of protective clothing as determined by the SHSO and the HSM. Access control points and delineation of entry/egress stations will be indicated on the Site Work Zone Map developed at the time of the on-site briefing conducted by the SHSO.

Prohibited items or conduct in the exclusion zone shall include, but not be limited to, the following:

1. Eating, drinking, smoking or any other activity which could lead to the possibility of hand to mouth exposure of contaminants;
2. Personal articles, *e.g.*, watches, rings, bracelets;
3. Working when ill, or taking prescription medication without HSM prior approval; and
4. Access to the exclusion zone by any individual not having the required health and safety training in accordance with 29 CFR 1910.120.

12.2 CONTAMINATION REDUCTION ZONE

The contamination reduction zone is established outside the exclusion zone and provides a transition between the exclusion zone (potentially contaminated zone) and the support zone (clean zone). It serves as a buffer to reduce the possibility of the support zone becoming contaminated and provides additional assurance that the physical transfer of contaminating substances on personnel and equipment or in the air is limited through a combination of decontamination, distance between exclusion and support zones, air dilution, zone restrictions, and work functions.

Items and/or conduct prohibited in the exclusion zone are also prohibited in the contamination reduction zone. Decontamination stations will be established at the boundary between the exclusion and the contamination reduction zones, for the sole purpose of personnel and equipment decontamination.

12.3 SUPPORT ZONE

The support zone shall be marked and protected against contamination from the work site. This will be accomplished by placing the support zone upwind of prevailing winds and providing adequate distance between the exclusion zone and the support zone. Primary functions of the support zone are:

1. The entry area for personnel, material, and equipment.
2. The exit area for decontaminated personnel, material, and equipment.
3. A storage area for clean safety and work equipment.
4. An area for rest breaks and the consumption of food and beverage, after washing of hands and face.

13.0 SAFE WORK PRACTICES AND EQUIPMENT

Employees and visitors will be required to follow and maintain good hygiene/work practices that include:

- Unauthorized personnel are not allowed on-site without prior briefing by the SHSO.
- Work groups will always consist of at least two team members; this may include AMEC and subcontract personnel.
- Smoking, eating, drinking, chewing gum or tobacco, taking medication, and applying cosmetics will not be permitted within any restricted areas or exclusion zone.
- Wearing of contact lenses in contaminated atmospheres is prohibited.
- Personnel under the obvious influence of alcohol or controlled substances are not allowed on-site. Those persons taken medication must notify the SHSO.
- Hands and face will be thoroughly washed before breaks or any hand to mouth activity such as eating, drinking, smoking, use of chewing tobacco, or application of cosmetics.
- Personnel will appropriately discard and replace any damaged, or heavily soiled protective clothing. Discarded PPE will be drummed at the end of each day.
- Personnel should notify the on-site health and safety coordinator of any defective monitoring, emergency, or other safety equipment.
- A supply of potable water, electrolyte replacement solutions, and sufficient lighting will be maintained on-site and accessible to personnel. The location of local sanitary facilities will be identified by the SHSO.
- All unsafe conditions shall be made safe immediately. All unsafe conditions not in the scope of the project shall be reported to the PM and the condition corrected.
- All site personnel will familiarize themselves with these and the emergency procedures during daily tailgate and prework safety meetings.

- Following safe work practices reduces the likelihood of an accident, illness, or injury during field activities.
- Loose-fitting clothing or loose long hair are prohibited near moving machinery.
- Workers who are passengers or drivers of vehicles (both offsite and onsite) will wear their seat belts any time the vehicle is in motion.
- Do not fuel engines while vehicle is running.
- Install adequate onsite roads, signs, lights, and devices.
- Store tools in clean, secure areas so they will not be damaged, lost, or stolen.

The HSM shall determine the types of safety and emergency equipment needed for the various tasks at the site as well as the necessary locations for the equipment (boat, field vehicle, etc.). This equipment may include, but not be limited to, a fire extinguisher, emergency eyewash, first aid kits, and oral thermometer (for measuring temperature associated with potential heat stress).

14.0 PERSONAL PROTECTIVE EQUIPMENT

Personal protective equipment (PPE) selection will be based on three criteria: U.S. Environmental Protection Agency (USEPA) Levels of Protection as defined in Standard Operating Safety Guides, the Occupational Health and Safety Guidance Manual for Hazardous Waste Site Activities, and the provisions of OSHA 29 CFR 1910 and 1926. In the event of conflicting requirements, the most stringent level shall apply. The anticipated level of protection for aquatic sampling activities is Level D and Level C for terrestrial sampling activities. These requirements are subject to change based on contaminant monitoring, visual observations, or changes in the work or site conditions. For tasks not covered in the HASP, personnel shall wear a level of PPE which is deemed necessary by the SHSO.

PPE will be selected, upgraded, or downgraded by the SHSO, based upon the specific task and the results of environmental monitoring and will be noted in the field notebook (Table 8). Employees will be provided with the appropriate protective equipment including chemical-resistant outer gloves, hard hats, safety shoes or heavy rubber shoes, and protective eye wear for field use. Rain gear will be available in the case of inclement weather. Employees will inspect and maintain all PPE as appropriate. All PPE for site activities is based upon the potential for contact with site-specific contaminants and may involve re-assessment necessitating upgrade or downgrade in PPE. The following outlines the requirements for each level of protection.

Four levels of protection and associated PPE are identified by OSHA for hazardous waste site work: Levels D, C, B, and A. Modified Level D (Level C, without a respirator) may also be used. Level D provides the lowest level of protection. Protection increases progressively through Levels C, B, and A. Level D is implemented unless a higher level of protection is specifically indicated. It is prudent to have available on-site, sufficient PPE to upgrade to the next higher level (*i.e.*, if level D is specified, Level C PPE should be available in case unforeseen hazards are encountered).

14.1 LEVEL D PPE

Most initial field activities require Level D if direct handling of contaminated materials is not anticipated or expected. If field conditions indicate a higher level of contamination than anticipated, a higher level of protection will be directed. If higher levels are necessary, they will be selected by the SHSO and/or HSM based on the contaminant concentrations. Level D requires minimal decontamination, including thorough washing of hands, arms, and face and boots, followed with safe work practices. Level D PPE includes the following:

1. Work shirt and work pants.
2. Safety toe work boots.
3. Hard hat (as necessary for required activity).
4. Approved safety glasses (with side shields) (as necessary for required activity)

14.2 MODIFIED LEVEL D PPE

Modified Level D includes:

1. Same as above but including the following:
2. Tyvek suits, Saranex or PE Tyvek may be required in wet conditions.
3. Safety toe, nitrile rubber high top boots (taped to Tyvek) or boot covers.
4. Inner vinyl gloves and outer nitrile rubber gloves (taped to Tyvek).
5. Face shield may be required under certain conditions (*i.e.*, splash protection).

14.3 LEVEL C PPE

The criteria for selection of Level C includes:

1. Oxygen level is greater than 19.5%.
2. Total unknown organic vapor levels at a sustained level of 5 ppm but below 50 ppm above background.
3. Doubt exists about the air quality, therefore preventing the use of Level D PPE.
4. Level A or B PPE is not indicated.

Level C PPE includes:

1. PPE same as Modified Level D.

2. Full-face air-purifying respirator with combination organic vapor/HEPA particulate (GMC-H) cartridges (or as otherwise designated); refer to AMEC respiratory protection program: SOP HSP-4.

14.4 LEVEL B PPE

The criteria for selection of Level B PPE includes:

1. Atmosphere with chemical concentrations considered IDLH.

2. Atmospheres exceeding the limits of the protection afforded by a full-face, air-purifying respirator.

3. Atmospheres containing substances with poor warning properties, substances for which air-purifying cartridges do not exist or have low removal efficiency.

4. Atmospheres containing less than 19.5% oxygen.

5. Conditions are such that small exposed areas about the head and neck will not be contacted by hazardous substances.

Level B PPE includes:

1. Same PPE as Level C, but with supplied air respirator (SCBA or airline with escape bottle); refer to AMEC respiratory protection program: SOP HSP-4.

All personnel will be required to be in appropriate PPE before entering the work zone. All disposable supplies shall be removed and disposed of in containers provided in the specified decontamination area. Personnel will be required to remove excess mud or other debris from boots and equipment before leaving the decontamination area and entering field vehicle.

15.0 PROTECTIVE CLOTHING DONNING/DOFFING PROCEDURE

The purpose of the protective clothing donning/doffing procedures is to ensure that on-site personnel are instructed in the proper way to don/doff protective clothing. Failure to adhere to these procedures may result in the protective clothing being ineffective against a potential contaminant. The following donning/doffing procedures are given as a guide and may be altered by the SHSO if improvements can be made to the procedure and these changes are warranted in the field. In addition, some articles of protective clothing/equipment detailed below may not be necessary for the particular site task.

15.1 PROTECTIVE CLOTHING DONNING PROCEDURE

1. Remove personal outer clothing in support zone and dress out in supplied clothing and rubber boots. Inspect clothing and respiratory equipment before donning.

2. Don Tyvek or splash suit, inner gloves, and outer rubber gloves as specified for the assigned task in Section 6.1. Outer chemical resistant gloves shall be sealed to the Tyvek suit using duct tape.

3. For the terrestrial sampling, don personal full-face respirator and cartridges by tightening straps. Test for fitness by completely covering cartridges and breathing in. No air should come into the respirator.

15.2 PROTECTIVE CLOTHING DOFFING PROCEDURES

1. Wash/rinse (if necessary) excess mud or other debris from outer boots, gloves, and clothing prior to leaving the exclusion zone.

2. Remove outer tape and outer layer of clothing (Tyvek suites, boots, gloves), placing disposable PPE in designated drums and reusable PPE in designated locations for donning during re-entry.

3. Remove respirator and discard cartridges prior to removing inner vinyl gloves. Remove inner gloves and move from the contamination reduction zone. Thoroughly decontaminate respirator with the manufacturer's approved detergent then store the

respirator in a protective plastic bag, which will be kept with worker or at appropriate storage facility in the support on-site at all times when not in use. Cartridges must be removed and disposed of properly at a hazardous material; do not store with the respirator.

4. Thoroughly wash hands and face with soap and water prior to eating, drinking, smoking, other hand-to-mouth contact activity.

16.0 DECONTAMINATION AND DISPOSAL

The purpose of decontamination is to prevent contaminants that may be present on protective clothing and equipment from coming into contact with personnel as they remove contaminated PPE. Also, decontamination protects workers from hazardous substances that may contaminate and eventually permeate the PPE used on-site; it protects personnel by minimizing the transfer of harmful materials into clean areas. Decontamination consists of physically removing contaminants or changing their chemical nature to innocuous substances. Combining decontamination with the correct sequential method of removing PPE will prevent exposure to personnel leaving the work areas as well as off-site migration of contaminants.

Generally, decontamination is accomplished by starting at the first station with the most heavily contaminated item and progressing to the last station with the least contaminated item. Each item of protective equipment requires a separate station which is marked accordingly.

The purpose of equipment decontamination is to prevent exposure to personnel during loading, transporting, and unloading at another site. It is also to prevent off-site migration of contaminants from one site to another or during transportation of the equipment.

Waste materials shall be disposed of to prevent the spread of contamination, creating a sanitary hazard or causing litter to be left on site. Disposal of potentially contaminated materials shall involve the bagging or drumming of the materials as necessary and segregation for special disposal. In addition, all non contaminated materials shall be collected and bagged for proper disposal as normal domestic waste.

16.1 PERSONNEL DECONTAMINATION

Removal of loose mud or other substrate from personnel will be performed in the decontamination zone. Personnel will remove any disposable PPE and dispose of it in provided containers before leaving the contamination reduction zone. Personnel shall thoroughly wash hands and face before leaving the area. A shower is not anticipated for use on-site at this time for personal decontamination. PPE and decontamination waste water will be collected and appropriately disposed.

16.2 EQUIPMENT DECONTAMINATION

All equipment (*e.g.*, hand tools, sampling equipment) shall be decontaminated at the job site prior to removal from the decontamination zone. Equipment decontamination will be conducted in accordance with USEPA protocols.

17.0 EMERGENCY RESPONSE

The purpose of this section is to safeguard human health and the environment in the event of an emergency. This section also addresses the emergency actions to be taken in response to an emergency. The responsibility of the regular day-to-day implementation of this information primarily lies with the SHSO. During an actual response situation, the SHSO will serve as the Emergency Coordinator.

17.1 PRE-EMERGENCY PLANNING

The SHSO will perform the following pre-emergency tasks before starting field activities and will coordinate emergency response with all appropriate personnel:

1. Locate nearest cellular telephone.
2. Confirm emergency telephone numbers (these numbers will be available on-site in this document for reference).
3. Inform Solutia and the local municipality or jurisdiction of the nature of the project hazards and potential emergencies.
4. Review and revise emergency response plan in the event of a failure of the plan in an emergency due to changes in site conditions, changes in the scope of work, or personnel availability.
5. Inventory and inspect on-site emergency equipment and supplies.

17.2 LINES OF AUTHORITY

The SHSO has primary responsibility for expediting Response Operations on-site to include reporting to the Ecological Project Manager and Solutia and correcting as conditions allow, and responding to and correcting emergency situations. The SHSO has the authority to stop any site activities posing an immediate health and safety hazard to site personnel and the public. Possible actions may involve notification of the HSM, the Ecological Project Manager, and Solutia, ensure corrective measures are implemented; notification of appropriate authorities; and follow-up reporting.

17.3 Emergency Prevention And Recognition

Prevention of emergencies will be aided by the effective implementation of the HASP, personnel awareness, contingency planning, and the briefing held with personnel at the beginning and during the execution of the field activities. Site security will be maintained during working hours by the SHSO. Communication is available by means of a cellular telephone or local phone service at neighboring commercial/retail establishments.

17.4 Notification

In the event of an emergency, a verbal instruction or site alarm will be sounded to:

1. Notify all on-site personnel. If appropriate, direct all personnel in the affected area to evacuate and assemble upwind in a designated safe area.
2. Stop work activities and shut down all combustion equipment
3. Establish the safety of all personnel, direct the administration of first aid and provide emergency equipment as appropriate.
4. Lower noise levels to facilitate communications.
5. Begin emergency procedures.
6. Notify on-site emergency response personnel about the emergency.
7. Prohibit outside personnel from entering the evacuated area until the Fire Department arrives.

Accident reporting should be performed in accordance with AMEC SOP HSP-1.

17.5 EVACUATION ROUTES AND PROCEDURES

In the event of an emergency that requires an evacuation of the site, an alarm will be sounded or verbal instruction given by the SHSO to evacuate the area. Personnel will exit the area to the specified meeting point. At this point, the SHSO will account for all personnel, ascertain information about the emergency and advise further instructions to the on-site personnel. The SHSO will also advise responding off-site emergency personnel of the situation, if necessary. In all situations that require evacuation, personnel shall not re-enter the work area until the conditions causing the emergency have been corrected, the hazard reassessed, the HASP has been revised if necessary and reviewed with on-site personnel, and instructions given for authorized re-entry.

17.6 EMERGENCY MEDICAL TREATMENT AND FIRST AID

In the event of an emergency involving personal injury or illness, first aid should be rendered by a trained person and emergency medical services summoned as identified in Section 17.9. Personnel with injury or illness will be decontaminated to the extent possible without further injury. Life saving and first aid procedures take priority over personnel decontamination efforts. The SHSO will have final authority on the decision to require additional professional medical services (*i.e.*, paramedics, hospital visit, etc.) for any illness or injury. In the case that emergency assistance is needed, the SHSO will immediately:

- Notify emergency response (911 or other numbers listed in the emergency contacts) and give the appropriate patient information and their location
- Assist the injured party as deemed appropriate
- Designate someone to accompany the injured party to the hospital

Accident reporting shall be performed in accordance with AMEC SOP HSP-1.

In the event that an exposure to toxic or hazardous materials occurs the first responder to the victim shall, as appropriate:

- Wash/rinse the effected area thoroughly with copious amounts of soap and water, then provide appropriate medical attention. If eyes are involved, they should be rinsed for at least 15 minutes using eyewash.

- Move the exposed person to fresh air and provide medical attention.

- Provide medical attention for ingestion and puncture wound or laceration.

17.7 Fire Or Explosion

In the event of a fire or explosion; the local fire department will be summoned immediately (Section 17.9). Upon their arrival, the SHSO will advise the fire commander of the situation. If it is safe to do so, on-site personnel may use fire fighting equipment available to control and/or extinguish the fire and remove or isolate flammable or other hazardous materials which contribute to the fire or inhibit control of the fire.

17.8 Spill Or Leak

In the event of a spill or leak (regardless of quantity), on-site personnel will:

1. Inform the HSM and Solutia PM immediately.

2. Locate the source and stop the spillage if it can be done safely.

3. Begin containment and recovery of spilled material.

17.9 EMERGENCY CONTACTS

Emergency resources are as follows:

Local Police Department:	Sauget	618-322-6507/6997
	Cahokia	618-337-5080
Local Fire Department:	Sauget	618-332-6700
	Cahokia	618-337-5080
Local Hospital:	St. Mary's Hospital	
	100 North 8th St.	
	East St. Louis, IL	618-274-1900
Poison Control Center:		1-800-942-5969
USEPA National Response Center:		1-800-424-8802

AMEC Contacts

Ecological Project Manager:	Chuck Harman	732-302-9500, ext. 127
Project Health and Safety Manager:	Jeffrey Tasca	732-302-9500, ext. 106
Corporate Health and Safety Manager:	Denise Daggett, CIH	858-458-9044

Sauget Area 2 Group Contact

Project Manager:	Steven Smith	314-674-4922

17.9.1 Route To Nearest Hospital

Turn right on Mobile Street then turn slight right onto Monsanto Ave. Continue on Monsanto Ave. for 0.3 mile. Turn left onto IL-3 for 0.9 mile. Take the I-70E/I-64E/I-55N ramp towards IL-3N/Chicago/Indianapolis and stay right at the fork in the ramp. Merge onto South 4th St. Turn right onto East Broadway/IL-15 and continue for 0.3 mile. Turn left on North 8th St, the hospital is #100. The route to the hospital is also indicated on the map contained in Attachment E.

17.9.2 Emergency Equipment

It shall be the responsibility of the AMEC SHSO to maintain the site emergency equipment in good working order. Equipment shall be readily available, clearly identified by signs and/or labels, and workers shall be trained in its use by the SHSO. Equipment maintained by the SHSO in the field vehicle and boat shall include a first aid kit, and an emergency eye wash kit. The boat is equipped with a fire extinguisher, signaling devices (e.g., air horn, lights) and a cell phone.

18.0 STANDARD PROCEDURE FOR REPORTING EMERGENCIES

The following information should also be provided to the Corporate Health and Safety and Personnel offices in the event of an emergency.

1. Name of person making call.
2. Telephone number at location of person making call.
3. Name of person(s) exposed or injured.
4. Information provided in medical data sheet (Attachment A).
5. Nature of emergency.
6. Actions already taken.

Specific accident reporting procedures are contained in AMEC SOP HSP-1.

18.1 FOLLOW-UP AND DOCUMENTATION

Before normal activities are resumed, on-site personnel must be prepared and equipped to handle another potential emergency. The follow-up activities should be completed:

1. Notify appropriate government agencies as required (Reminder: OSHA must be notified within eight hours if there have been any fatalities or three or more hospitalizations).
2. Restock all equipment and supplies.
3. Review and revise all aspects of the HASP as necessary to address future emergencies of this type and new site conditions.

Investigation and documentation of any emergency response shall be initiated by the Field Team Leader with assistance by the SHSO. This is important in all cases, but especially so when the incident has resulted in personal injury, property damage, or environmental impact. The documentation will be a written report and will be:

1. Accurate: All information must be recorded objectively.

2. Authentic: Each person making an entry must sign and date that entry. Nothing is to be removed or erased. If details are changed or revised, the person making the change should strike out the old material and initial and date the change.

3. Titles and names of personnel involved.

4. Actions taken, decisions made, orders given, to whom, by whom, when, what, where, and how as appropriate.

5. Summary of data available (air monitoring, chemical concentrations, etc.).

6. Possible exposure of personnel.

7. Copies of all the Supervisor's Accident Investigation Reports and Employer's Report of Occupational Injury or Illness.

19.0 PROJECT DOCUMENTATION

Site investigation information shall be documented with the use of the reporting forms and logs shown in this section. The logs and records shall be the ultimate responsibility of the SHSO and shall be maintained on-site for review. However, all personnel shall be individually responsible for completion of the information required by the logs. The health and safety logs and records, included as attachments and summarized below, shall be kept on record for a period of no less than 10 years by AMEC.

Plan Approval Signature Sheet	Section 20.0
Site Map	(Figure 2, URS Figure 1)
Daily Project Log	Field Notebook
Site Monitoring Data	Field Notebook
Training/Safety Briefing Log	Field Notebook
Accident/Illnesses Reports	SOP HSP-1 Forms

Record keeping for health and safety purposes will be in accordance with the OSHA requirements

published in 29 CFR 1910, including medical examination reports, accident/injury reports, exposure reports, and records/certificates of the general and specialized training courses completed by each person employed on-site.

20.0 PLAN APPROVAL

This HASP has been written for the use of AMEC employees and subcontractors on this project. AMEC claims no responsibility for its use by others. The HASP is written for the specific site conditions, purposes, dates and personnel specified and must be amended if these conditions change.

Jeffrey Tasca

Project Health and Safety Manager

Charles Harman, PWS

Project Manager

Site Health and Safety Officer

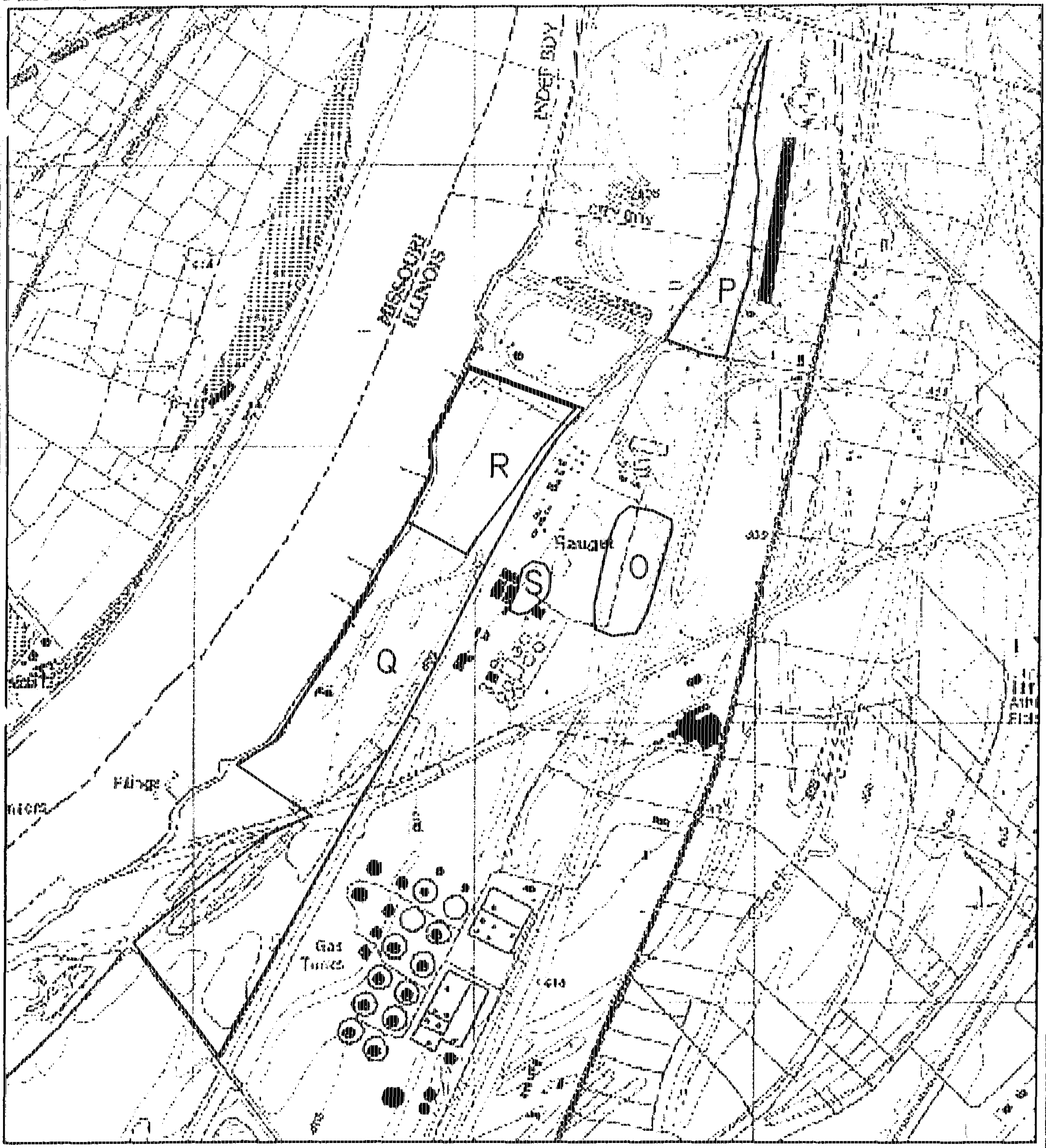
TBD

21.0 HEALTH AND SAFETY PLAN ACCEPTANCE

I have had the opportunity to read and ask questions about this HASP. My signature certifies that I understand the procedures, equipment, and restrictions of this plan and agree to abide by them.

SIGNATURE*	PRINTED NAME	COMPANY	DATE

This acceptance form is required for all routine site staff and subcontracting personnel. Visitors and non-routine subcontractors are required to receive and sign the appropriate Health and Safety Orientation Form located in Attachment F.



SOURCE: USGS QUADRANGLE (CAHOKIA, IL–MO), 1998
NOT TO SCALE

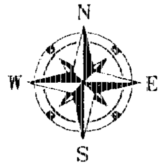
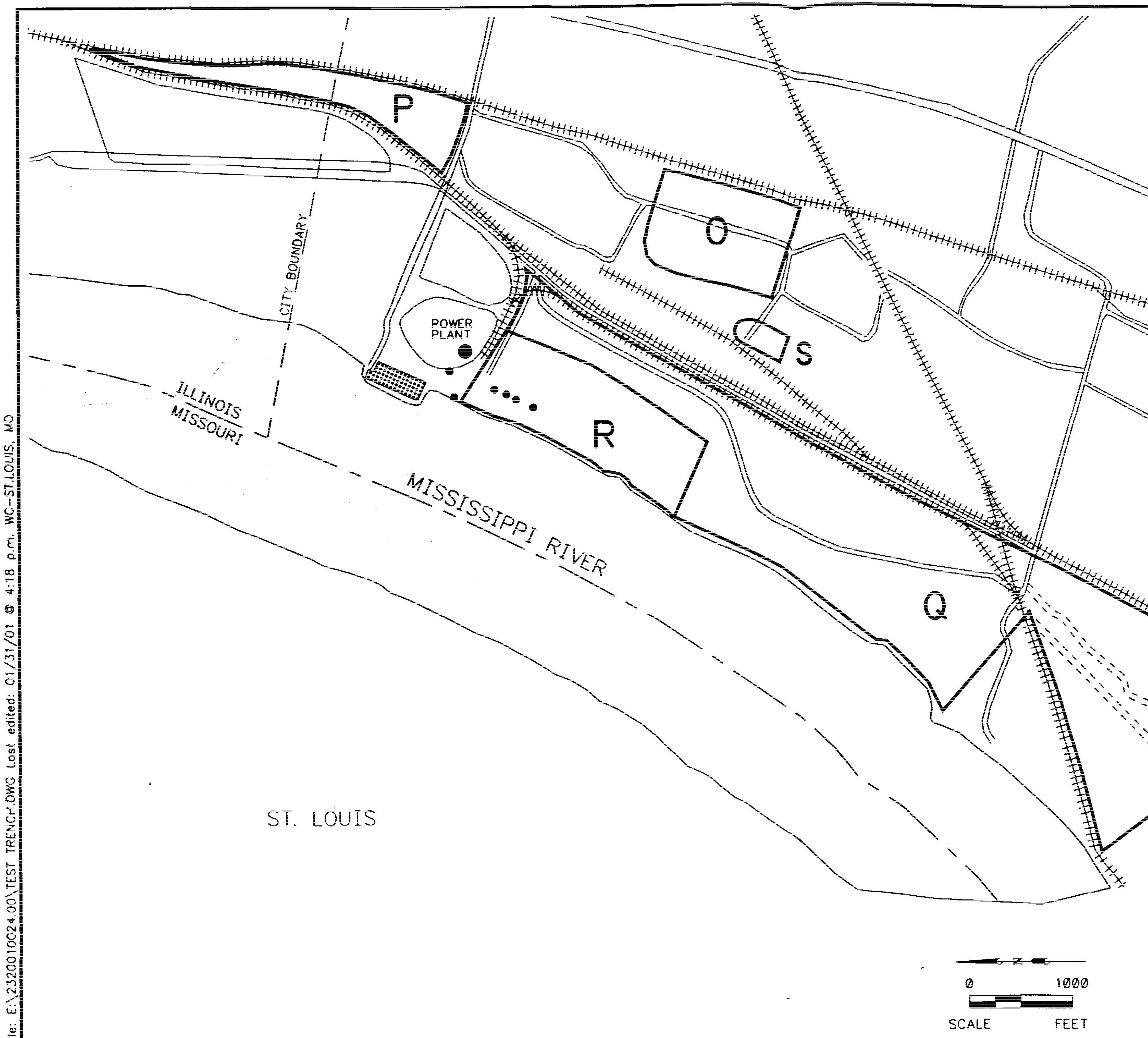


FIGURE 1

SITE LOCATION MAP

SAUGET AREA 2
SITES O, P Q, R, S
SAUGET AND CAHOKIA, ILLINOIS



SAUGET AREA 2 RI/FS SAMPLING LOCATIONS
SAUGET ILLINOIS

PROJECT NO. 2320010024.00

URS

DRN. BY: djd 1/31/01
DSGN. BY: bv
CHKD. BY:

Site Vicinity Map

FIG. NO. 1

File: E:\2320010024.00\TEST TRENCH.DWG Last edited: 01/31/01 @ 4:18 p.m. WC-ST.LOUIS, MO

Table 1
Maximum Constituent Concentrations in Soil
Sauget Area 2

Constituent	Site O	Site P	Site Q	Site R [A]	Site S
VOCs				5800	
benzene	30.8				
chlorobenzene	58.9		100		
ethylbenzene	167		790		450
4-methyl-2-pentanone	7.69		250		93
toluene	29.5	0.413	2400		990
1,1,1-trichloroethane	1.41				12
o-xylene			2300		
xylenes (total)	615.4	0.45			620
SVOCs				19000	
1,4-dichlorobenzene	1030	8.87	1200		
1,2-dichlorobenzene	606	3.625			
1,2,4-trichlorophenol	26.9				
naphthalene	34.6				200
2-methylnaphthalene	160				
N-nitrosodiphenylamine	50				
pentachlorophenol	1620				
phenanthrene	230				
fluoranthene	74				
pyrene	282				
butyl benzyl phthalate	3846				490
benzo(a)anthracene	121.7				
1,2,4-trichlorobenzene	65.3				
chrysene	282				
phenol		3.875			
di-n-butyl-phthalate		16.25	900		1500
di-n-octyl-phthalate					310
bis(2-ethylhexyl)phthalate			1100		20000
PCBs				4800	
Aroclor 1232	30.3				
Aroclor 1242	1871				
Aroclor 1248			70		85
Aroclor 1254			360		69
Aroclor 1260			16000		41
Dioxins					
2,3,7,8-TCDD	0.00017		0.0033		
Metals					
antimony			17900		
arsenic			0.216		
cadmium	31		152000		
chromium			3650		
copper	341		1630		139
cyanide		15			
lead		526	195000		0.392
mercury	6.3	3.9	4.9		3.5
nickel	136		371		
selenium			59.9		
silver			30.2		
thallium			0.89		
zinc	1398		9520		327

all concentrations in ppm

A - Soil sampling at Site R showed VOC concentrations ranging from .15 to 5800 ppm. SVOCs were found at levels ranging from 0.017 to 19,000 ppm. Pesticides were found at levels ranging from 0.11 to 99 ppm and PCBs were detected at levels ranging from 0.75 to 4,800 ppm. Elevated levels of arsenic, chromium, lead, nickel, and mercury were also detected in Site R soils.

Table 2

Project Organization and Roles

NAME	PROJECT ROLE	PHONE NUMBER
Sauget Area 2 Sites Group		
Steven Smith	Project Manager	314-674-4922
AMEC Earth and Environmental, Inc.		
Charles Harman, PWS	Project Manager	732-302-9500
Laurie Gneiding, CEP	Terrestrial Field Team Leader	732-302-9500
J. David Dean	Aquatic Field Team Leader	770-420-2100
TBD	SSHO	
Jeffrey Tasca	Project H &S Manager (HSM)	(732) 302-9500
Denise Daggett, CIH	Corporate H &S Officer	(858) 458-9111

Table 3
Activity Hazard Analysis

Project Identification Sauget Area 2	Location Sauget and Cahokia Illinois	Estimated Start Date October 2001
TASK	**POTENTIAL HAZARDS**	**CONTROL MEASURES**
Aquatic Sampling (surface water, sediment and biota collection)	Exposure to Chemical Hazards	* Wear appropriate PPE * Practice contamination avoidance * Follow proper personal and sample decontamination procedures. * Wash hands/face immediately as part of decontamination * Wear chemical safety goggles when handling chemical sample preservatives and samples * Avoid splashing. If inevitable, personnel should stay out of splash radius. * Wear chemical protective gloves (nitrile).
	Manual Lifting and Material Handling	* Use proper lifting techniques * Team lifting will be used for heavy loads (> 60 lbs.)
	Cold Stress	* Personnel must be aware of sign/symptoms * Personnel must drink plenty of fluids * Practice cold stress prevention per HSP
	Splashing	* Use safety glasses or goggles; and * All personnel should stay out of the splash radius.
	Slips/Trip/Falls	* Work areas and means of access shall be maintained neat and orderly * Even terrain will be utilized as unloading areas
	Boating Operations	* Individuals operating boasts must be experienced and qualified * Boats are to be occupied during use by not less than one qualified operator plus one additional person. * The designated boat operator will provide a safety briefing to all boat occupants prior to disembarking * Maximum weight load for a boat will not exceed manufacturer's specified capacity. * All persons on board will remain seated except when sampling * All gear will be stowed securely against unexpected shifts. * All personnel on board will wear a Coast Guard approved Type II personal floation devices. * On-board personnel must be able to contact shore either by cellular phone or radio
Terrestrial Sampling (vegetation and macroinvertebrate sample collection)	Exposure to Chemical Hazards	* Wear appropriate PPE per HASP * Practice contamination avoidance * Follow proper personal and sample decontamination procedures * Wash hands/face immediately as part of decontamination * Wear chemical safety goggles when handling chemical sample preservatives and samples * Avoid splashing. If inevitable, personnel should stay out of splash radius * Hazard communication training
	Manual Lifting, Material Handling, and Hand Auger Usage	* Use proper lifting techniques * Team lifting will be used for heavy loads (> 60 lbs.)
	Cold Stress	* Personnel must be aware of sign/ symptoms of cold stress * Personnel will drink plenty of fluids * Practice cold stress prevention per HASP
	Slips/Trip/Falls	* Work areas and means of access shall be maintained neat and orderly * Even terrain will be utilized as unloading areas

(ıble 4
Chemical Hazard Properties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
Aniline	5 ppm	Ca (100 ppm)	ND	11%/1.3%	Inh Con	Headache, weakness, dizziness, fast heartbeat, cyanosis[7]; eye irritant	colorless to brown, oily liquid with aromatic amine-like odor. Solid below 21°F	strong oxidizers, strong acids, toluene diisocyanate, alkalis
Antimony	0.5 mg/m^3	50 mg/m^3	NA	NA/NA	Inh Con	Irritant to eyes, skin, nose, throat, and mouth. Coughing, dizziness, headache, and loss of olfactory function (smell).	Silver-white, lustrous, hard, brittle solid. Scale like crystals or a dark-gray lustrous powder.	strong oxidizers, acids, halogenated acids.
Arsenic (inorganic compounds)	0.01 mg/m^3 / 0.5 mg/m^3	0.002 mg/m^3/ 5 mg/m^3	NA	NA/NA	Inh Abs Con	Ulceration of nasal septum, dermatitis, respiratory irritation, darkening of skin, [carc]	Metal: Silver-gray or tin-white brittle, odorless solid.	Strong oxidizers, bromine, azide
Benzene	0.5 ppm/1 ppm	1 ppm/ 500 ppm	9.24	1.2%/7.7.8 %	Inh Abs Con	Irritation oft eyes, skin, nose, respiratory system; giddiness; headache, nausea, staggered gait; fatigue, anorexia, tiredness; dermatitis; [carc]	Colorless to light-yellow liquid with an aromatic odor. [Note: A solid below 42° F.]	Strong oxidizers, fluorides, perchlorates, nitric acid
Bis(2-ethylhexyl)phthalate	5 mg/m^3	10 mg/m^3/ 5000 mg/m^3	ND	0.3%/ND	Inh Con	Irritant to eyes, mucous membranes.	Colorless, oily liquid with a slight odor.	
Butyl benzyl phthalate	ND	5 mg/m^3/ ND	ND	ND	Inh Con	ND	clear, oily liquid	Oxidizers
Cadmium	0.01 ppm 0.005 mg/m^3	9 mg/m^3	NA	NA/NA	Inh Ing	Fluid in lungs, difficulty breathing, coughing, chest tightness, headache, chills, muscle aches, nausea, vomiting, diarrhea, mild anemia, [carc]	Metal: Silver-White, blue-tinged, lustrous, odorless solid.	strong oxidizers, sulfur, selenium, tellurium

NA: Not applicable
ND: No data available
[1] OSHA PEL = OSHA Permissible Exposure Limit is a promulgated concentration that cannot be exceeded during an 8-hour time shift over a 40-hour work week.
[2] ACGIH TWA = Guideline not to be exceeded during an 8-hour time shift over a 40-hour work week.
[3] STEL = short-term exposure limit. Concentration not to be exceeded for 15-minute time-weighted average.
[4] IDLH = *immediately dangerous to life and health. Represents the maximum concentration from which, in the even of respirator failure, one could escape within 30 minutes without a respirator* and without experiencing any escape-impairing or irreversible health effects.
[5] IP = ionization potential. Given as a guideline to determine which lamp to use with a photoionization detector (PID).
[6] LEL/UEL = lower explosive limit/upper explosive limit. Represents the upper and lower flammable limits in air at room temperature.
[7] Cyanosis = purple discoloration of skin due to lack of oxygen

(able 4
Chemical Hazard Properties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
Chloroaniline	5 ppm/ 2 ppm	ND	ND	2.2/ND	Inh Con	Cyanosis, headache, redness of skin, eye irritant, nausea, vomiting, weakness, convulsions	colorless to yellow crystals	Strong oxidizers, acids, acid chlorides, acid anhydrides
Chlorobenzene	10ppm 750 ppm	1000 ppm	9.07	1.3%/9.6%	Inh Con	Irritated eyes, skin, and nose. Drowsiness and incoherence.	Colorless liquid with an almond like odor.	strong oxidizers
Chlorophenol	ND	ND	ND	ND	Inh Con	Cough, dizziness, headache, labored breathing, sore throat; skin redness; eye irritant; thickening of skin	colorless crystals with characteristic odor.	Oxidants
Chromium (as Cr^{3+} dust)	0.5 mg/m^3 1 mg/m^3	25 mg/m^3	NA	NA/NA	Inh Con	Irritant to eyes, skin, and lungs	Blue-white to steel-gray, lustrous, brittle, hard, odorless solid.	compound specific
Copper (dusts)	1 mg/m^3 1 mg/m^3	100 mg/m^3	NA	NA/NA	Inh Con	Irritant to eyes, nose, pharynx, and nasal passages.	Reddish, lustrous, malleable, odorless solid.	oxidizers, alkalis, acetylene
Cyanide	10 ppm	50 ppm	13.6	5.6%/40%	Inh Abs Con	Asphyxiation, weakness, headache, confusion, nausea, vomiting, increased rate and depth of respiration, or slow and gasping respiration.	Colorless or pale blue liquid or gas above 78° F. Bitter, almond like odor.	
Di-n-butylphthalate	5 mg/m^3	4000 mg/m^3	ND	0.5%/ND	Inh Abs Con	Eye and upper respiratory tract irritant.	Colorless to faint yellow oily liquid	nitrates, strong oxidizers, alkalis, acids, liquid chlorine
Dichlorobenzenes (1,2-; 1,4)	1,2-: 50 ppm 1,4-: 75 ppm	1,2-: 200 ppm 1,4-: 150 ppm [Ca]	8.98	2.5%/NA	Inh Abs Con	Irritation to eyes, and nose. Liver and kidney damage. Skin blisters; headache	Colorless to pale yellow liquid with a pleasant aromatic odor./ Colorless or white crystalline solid with a mothball like odor.	
Dichlorophenol	ND	ND	ND	ND	Inh Con	Eye , skin, respiratory tract irritation	white-brown crystals	Oxidizers, acid chlorides, acid anhydrides
Di-n-octylphthalate	5 mg/m^3	10 mg/m^3/ ND	ND	0.3%/ND	Inh Con	Respiratory tract, skin and eye irritant	Light-colored, colorless liquid	Strong oxidizers, acids, and alkalis
Ethyl benzene	100 ppm/ 100 ppm	125 ppm/ 800 ppm	8.76	0.8%/ 6.7%	Inh Con	Irritant to eyes, skin, mucous membranes; headache; dermatitis, narcosis (tiredness), coma	Colorless liquid with an aromatic odor.	Strong oxidizers

(ble 4
Chemical Hazard Propeı ties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
Isopropanol (70%)	400 ppm 400 ppm	2000 ppm	10.10	2.0%/12.7 %	Inh Con	Irritation eyes, nose, throat; drowsiness, dizziness, headache; dry cracking skin;	Colorless liquid with the odor of rubbing alcohol.	Strong oxidizers, acetaldehyde, chlorine, ethylene oxide, acids, isocyanates
Lead	0.1 mg/m^3 0.05 mg/m^3	100 mg/m^3	NA	NA/NA	Inh Con	Weakness, insomnia, facial pallor, irritated eyes, and hypotension.	A heavy, ductile, soft, gray solid.	Strong oxidizers, hydrogen peroxide, acids
Mercury (except organomercury)	Hg vapor: 0.05/0.1 mg/m^3 Other: 0.1 mg/m^3	10 mg/m^3	NA	NA/NA	Inh Abs Con	Irritation to eyes, and skin. Coughing, chest pain, difficulty breathing, bronchitis, tremors, insomnia, irritability, indecision, headache, fatigue, weakness	Metal: Silver-white, heavy, odorless liquid.	acetylene, ammonia, chlorine dioxide, azides, calcium, sodium carbide, lithium, copper
4-Methyl-2-pentanone	50 ppm 100 ppm	500 ppm	9.34	?/8%	Inh Con	Irritation to eyes, nose, and skin. Headaches, sleepiness, coma,	Colorless liquid with a pleasant odor.	
Nickel (metals or other compounds except nickel carbonyl)	0.015 mg/m^3 * 1 mg/m^3	10 mg/m^3	NA	NA/NA	Inh Con	Dermal sensitivity, allergic asthma, and pneumonitis[8] [carc]	Metal: Lustrous, silvery odorless solid	Strong acids, sulfur, selenium, wood and other combustibles.
N-nitrosodiphenylamine	ND	ND	ND	ND	Inh Con	Eye irritant, skin redness, skin edema, [Carc]	greenish crystals	Oxidizers
PAHs (as coal tar pitch volatiles)	0.2 mg/m^3 / 0.2 mg/m^3	ND/80 mg/m^3	ND	ND	Inh Con	Dermatitis, bronchitis [carc]	Black or dark brown amorphous residue	
PCBs	0.5 mg/m^3 1 mg/m^3	5 mg/m^3	ND	ND	Inh Abs Con	Irritation to eyes, chloracne, liver damage, reproductive effects. [carc]	Colorless to light colored viscous liquid with a slight hydrocarbon odor.	Strong oxidizers
Pentachlorophenol	0.5 mg/m^3 0.5 mg/m^3	2.5 mg/m^3	ND	ND	Inh Abs Con	Irritation to eyes, nose, throat. Sneezing, coughing, sweating, headache, dizziness, nausea, vomiting, difficulty breathing chest pain, high fever, dermatitis.	Colorless to white, crystalline solid with a benzene like odor. [fungicide]	Strong oxidizers, acids, alkalis

[8] Pneumonitis = inflammation of the lungs.

(ıble 4
Chemical Hazard Prope. ties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
Phenol	5 ppm 5 ppm	250 ppm	8.5	1.8%/8.6%	Inh Abs Con	Irritation to eyes, nose, and throat. Weakness, muscle aches, pain, dark urine, cyanosis , skin burns, dermatitis, ochronosis[9], tremors, convulsions, twitching.	Colorless to light pink crystalline solid with a sweet acrid odor.	Strong oxidizers, calcium hypochlorite, acids
Selenium	0.2 mg/m^3 0.2 mg/m^3	1 mg/m^3	NA	NA/NA	Inh Con	Irritation to eyes, nose, and throat. Vision disturbances, headache, chills, fever, bronchitis, dermatitis, skin burns	Amorphous or crystalline red to gray solid.	Acids, Strong oxidizers, chromium trioxides, potassium bromate, cadmium
Silver (metal dust and soluble compounds)	0.1mg/m^3 0.1 mg/m^3	10 mg/m^3	NA	NA/NA	Inh Con	Blue-gray eyes, nasal septum, throat, skin irritation, skin ulceration,	Metal; white lustrous solid	Ethyleneimine, acetylene, hydrogen peroxide, bromoazide, chlorine trifluoride
2,3,7,8-tetrachlorodibenzo-p-dioxin	ND	ND	ND	ND	Inh Abs Con	Irritation to eyes, skin, mucous membranes, thermal skin burns, headache, sore throat.	Colorless to white crystalline solid	
Thallium	0.1 mg/m^3 0.1 mg/m^3	15 mg/m^3	NA	NA	Inh Abs Con	Ptosis[10], strabismus[11], tremor, chest pain and tightness, pulmonary edema, seizures, uncontrolled movements, psychosis, hair loss, numbness in legs.	Appearance and odor vary depending upon the specific soluble thallium compound..	
Toluene	50 ppm [skin]	150 ppm/ 500 ppm	8.82	1.1%/ 7.1%	Inh Abs Con	Irritant to eyes, nose; fatigue, weak, confusion, euphoria, dizziness, headache; dilated pupils, tearing eyes; nervousness, muscle fatigue, insomnia; dermatitis	Colorless liquid with a sweet pungent, benzene-like odor.	Strong oxidizers
1,2,4-Trichlorobenzene	5 ppm	ND	ND	ND/6.6%	Inh Con	Irritation to eyes, skin, mucous membranes	Colorless liquid or crystalline solid (below 63 F) with an aromatic odor.	Acids, acid fumes, oxidizers, steam
1,1,1-Trichloroethane	350 ppm	700 ppm	11	7.5%/12.5 %	Inh Con	Irritation to eyes, and skin. Headache, lassitude, CNS[12] depression, poor equilibrium, dermatitis, cardiac arrhythmia: liver damage	Colorless liquid with a mild chloroform like odor.	ND

[9] Ochronosis = pigment deposits in the cartilages, ligaments, and tendons associated with alkaptonuria (alkaptons in urine).
[10] Ptosis = sagging organ or parts, e.g., drooping upper eyelid
[11] Strabismus = inability for one eye to focus for binocular vision
[12] CNS = central nervous system

(able 4
Chemical Hazard Properties and Exposure Information

CHEMICAL NAME/ SYNONYM	OSHA PEL[1]/ ACGIH TLV[2]	STEL[3]/ IDLH[4]	IP[5] (eV)	LEL/UEL[6]	RELEVANT EXPOSURE PATHWAY	SYMPTOMS	PROPERTIES/ CHARACTERISTICS	INCOMPATABILITIES/ REACTIVITIES
1,2,4-Trichlorophenol	ND	ND	ND	ND	ND	ND	ND	ND
Xylenes (o, m, p)	100 ppm/ 100 ppm	150 ppm/ 900 ppm	8.56 (o, 8.44(p)	m,p: 1.1%/7.0% o: 0.9%/6.7%	Inh Abs Con	Irritant to eyes, skin, nose, throat; dizziness, excitement, drowsiness, incoordination, staggering gait; dermatitis.	Colorless liquid with an aromatic odor.	Strong oxidizers, strong acids
Zinc (as zinc oxide)	5 mg/m³ * 5 mg/m³	10/500 mg/m³	NA	NA/NA	Inh Con	Irritant to eyes, skin, upper respiratory system: coughing.	White, odorless solid	water

TABLE 5

Wind Chill Factors

Wind Speed (mph)	Local Temperature, °F										
	32	23	14	5	-4	-13	-22	-31	-40	-49	-58
	Equivalent Temperature, °F										
Calm	32	23	14	5	-4	-13	-22	-31	-40	-49	-58
5	29	20	10	1	-9	-18	-28	-37	-47	-56	-65
10	18	7	-4	-15	-26	-37	-48	-59	-70	-81	-91
15	13	-1	-13	-25	-37	-49	-61	-73	-85	-97	-109
20	7	-6	-19	-32	-44	-57	-70	-83	-96	-109	-117
25	3	-10	-24	-37	-50	-64	-77	-90	-104	-117	-121
30	1	-13	-27	-41	-54	-68	-82	-97	-109	-123	-137
35	-1	-15	-29	-43	-57	-71	-85	-99	-113	-127	-142
40	-3	-17	-31	-45	-59	-74	-87	-102	-116	-131	-145
45	-3	-18	-32	-46	-61	-75	-89	-104	-118	-132	-147
50	-4	-18	-33	-47	-62	-76	-91	-105	-120	-134	-148
LITTLE DANGER FOR PROPERLY CLOTHED PERSONS		**CONSIDERABLE DANGER**			**VERY GREAT DANGER**						
Maximum danger of false sense of security		Danger from freezing of exposed flesh within one minute			Flesh may freeze within 30 seconds						
Trenchfoot and immersion foot may occur at any point on this chart											

TABLE 6
Sauget Area 2 Sites
Health and Safety Plan

WORK/WARM-UP SCHEDULE FOR FOUR-HOUR SHIFT

Air Temp. - Sunny Sky	**No Noticeable Wind**		**5 mph Wind**		**10 mph Wind**		**15 mph Wind**		**20 mph Win**	
°F (approx.)	**Max. Work Period**	**No. of Break s**	**Max. Work Period**	**No. of Break s**	**Max. Work Period**	**No. of Break s**	**Max. Work Period**	**No. of Break s**	**Max. Work Perio d**	**No. Brea**
-15° to -19°	norm.breaks	1	norm. breaks	1	75 min.	2	55 min.	3	40 min.	4
-20° to -24°	norm. breaks	1	75 min.	2	55 min.	3	40 min.	4	30 min.	5
-25° to -29°	75 min.	2	55 min.	3	40 min.	4	30 min.	5	Non-emergen work should cease ↓	
-30° to -34°	55 min.	3	40 min.	4	30 min.	5	Non-emergency work should cease ↓		↓	
-35° to -39°	40 min.	4	30 min.	5	Non-emergency work should cease ↓		↓		↓	
-40° to -44°	30 min.	5	Non-emergency work should cease ↓		↓		↓		↓	
-45° & below	Non-emergency work should cease		↓		↓		↓		↓	

NOTES:

1. Schedule applies to any 4-hour work period with moderate to heavy work activity, with warm-up periods in a warm location and with an extended break (e.g., lunch) a the end of the 4-hour work period in a warm locations. For light-to-moderate work (limited physical movement): apply the schedule one step lower.

2. TLV applies only for workers in dry clothing.

Source: ACGIH TLVs and BEIs. 1992/1993

Table 7
Type and Frequency of Air Monitoring

TYPE	MINIMUM RECOMMENDED FREQUENCY
Background:	Once per day in the work area and perimeter using direct-reading instruments, prior to any intrusive activities or equipment startup.
Perimeter:	Once per hour using direct-reading instruments during intrusive activities.
Personnel:	At least twice per day in the breathing zone of those with the highest anticipated exposure during intrusive activities.
Area:	At least twice per day in each work zone and at the onset of any new intrusive activities, or at new locations.
Environmental:	Periodic field screening of selected samples as per the Sampling and Analysis Plan.

Table 8
Action Levels for Change in PPE

EQUIPMENT	ACTION LEVEL	ACTION TO BE TAKEN
PID	>5 ppm sustained for greater than 5 minutes	Upgrade to level C
PID	>50 ppm	Upgrade to level B
PID	>500 ppm	Stop all work activities, evacuate the site and call proper authorities.

ATTACHMENT A

MEDICAL DATA SHEET

MEDICAL DATA SHEET

This brief Medical Data Sheet will be completed by all onsite personnel and will be kept in the support zone by the Site Health and Safety Officer during the conduct of the site operations. Completion of this form is required in addition to compliance with the Medical Surveillance Program requirements. This data sheet will accompany any personnel when medical assistance is needed or if transport to a hospital facility is required.

Project:		
Name:		Home Telephone:
Address		
Age:	Height:	Weight:
Blood Type:		
Emergency Contact:		
Drug or Other Allergies:		
Particular Sensitivities		
Do you wear contacts?		
Provide a checklist of previous illnesses		
Exposure to Hazardous Chemicals		
What medications are you presently using?		
Do you have any medical restrictions?		
Name/Address/Phone Number of Personal Physician		

ATTACHMENT B

INCIDENT REPORT

1.0 INTRODUCTION

Accident investigation is a useful tool for discovering the cause(s) of an accident and to prevent future accidents. A thorough investigation of the contributing circumstances that might have caused an accident (including both injury accidents and non-injury accidents [incidents]) is crucial. The real or potential extent of injury or property damage dictates the thoroughness of the investigation. Causative factors are then evaluated to determine the appropriate corrective action. Detailed corrective action information is provided in the Injury and Illness Prevention Program, Volume I, of this manual.

Accident investigation is not to be confused with injury or illness reporting (discussed in SOP A-3, *Injury or Illness Reporting*).

2.0 SCOPE

This procedure applies to both accidents which occur in office operations or field jobsites. This procedure pertains to accidents involving OEES personnel, personnel subcontracted to OEES, and visitors, including clients, who may work on or visit OEES jobsites.

OEES reserves the right to investigate and document any accident involving a subcontractor.

3.0 RESPONSIBILITIES

3.1 CORPORATE HEALTH AND SAFETY DIRECTOR (CHSD)

The CHSD manages an accident investigation, either parallel to or in conjunction with the Project Manager (PM)/Supervisor, as the circumstances warrant. In instances of a severe injury or fatality, or a potentially catastrophic near miss, the CHSD will investigate and report to the Executive Vice President of Operations. The CHSD serves in an advisory capacity for all accident investigations. The CHSD also periodically analyzes the accident investigation data for the purpose of identifying underlying causes and general patterns.

3.2 Health and Safety Manager (HSM)

The HSM is responsible for initiation and oversight of accident investigations. Depending on the breadth of the investigation, the HSM may conduct the accident investigation.

3.3 PM/Supervisor

The PM/Supervisor is responsible for conducting accident investigations relating to projects or activities under his/her supervision. The PM/Supervisor is also responsible for ensuring that corrective actions (identified through the accident investigation) are implemented within the targeted time frame and that the appropriate injury/illness or incident reports are made.

3.4 Health and Safety Coordinator (HSC)/Site Health and Safety Coordinator (SHSC)

The HSC/SHSC shall assist the PM/Supervisor during accident investigations and shall verify the PM/Supervisor's findings.

3.5 Employees and Subcontractors

Employees and subcontractor employees are responsible for cooperating with accident investigators during the investigation and for being truthful and answering questions to the best of their recollection. Employees and subcontractor employees shall also comply with corrective action recommendations within the targeted time frame.

4.0 GOALS OF ACCIDENT INVESTIGATION

The PM/Supervisor should make a personal investigation of all accidents because:

- An investigation is the best way to determine the true cause of an accident.
- The PM/Supervisor should know what the employee was doing, the proper way to do it, and what the employee probably did or did not do to cause the accident.
- The PM/Supervisor is best qualified to evaluate the information gathered and determine the cause of the accident.
- The PM/Supervisor has the authority to investigate the accident and initiate corrective action.

5.0 INVESTIGATIVE PROCEDURES

The PM/Supervisor shall conduct an investigation of all incidents, injuries, and work-related illnesses; talk with the victim and/or co-workers who witnessed the accident or conditions; and examine the doctor's medical report if available. Each investigation shall be conducted as soon after the accident/incident as possible. Delays of even a few hours can allow for the destruction/alteration of evidence (whether intentional or unintentional).

Fairness and impartiality are essential during the investigation. The purpose of the investigation is to obtain information and prevent a recurrence of the incident/accident, not to place blame.

5.1 REPORTING

The PM/Supervisor shall report verbally and in writing any injury, illness, or incident on the appropriate forms and to the appropriate parties as defined in SOP A-3.

5.2 INVESTIGATING

The investigation phase of the accident investigation procedure is initiated in response to reports of an accident (either injury or non-injury). Important information that should be obtained through the investigation include:

- Project
- Location of accident
- Employee(s) involved
- Narrative description of the accident
- Equipment associated with the accident
- Task being performed when accident happened
- Time factors (e.g., time of day, hours into shift, type of shift)
- Preventive measures
- Characteristics of injury or property damage

5.2.1 Interviewing

After reviewing the Supervisor's Report of Injury or Illness, First-aid Incident Report, and/or Incident Report, or after verbal report of an accident, the PM/Supervisor and HSC/SHSC shall interview individually all witnesses to the accident. It is recommended to prepare a set of questions and ask each witness to answer the same questions. The details of the interview shall be recorded on the Accident Investigation Interview Report (Attachment 1). The completed form shall be forwarded to the appropriate HSM, office HSC, SHSC, and CHSD.

5.2.2 Identifying Causal Factors and Corrective Actions

The PM/Supervisor shall identify contributing factors to the accident using the National Safety Council's *Guide for Identifying Causal Factors and Corrective Actions* (Attachment 2). Completed guides shall be forwarded to the appropriate HSM, office HSC, SHSC, and CHSD.

Possible corrective actions are suggested on the guide, but keep in mind that these are not the only possible corrective actions that may be implemented. The recommended (and mandatory) corrective action(s) will be decided upon by the PM/Supervisor, with the assistance of the HSC/SHSC and CHSD (when necessary), and stated in the appropriate column of the guide.

Corrective actions will be selected based upon effectiveness, cost versus benefit, feasibility, reliability, acceptance, effect on productivity, time required to implement, and any other factor deemed significant. Corrective actions can be monitored for implementation and completion using the Corrective Action Program Schedule (Attachment 3).

5.3 ANALYSIS

The CHSD periodically analyzes the accident investigation data for the purpose of identifying underlying causes or general patterns. The analysis will also identify inadequate policies, procedures, or management systems that are not always readily evident when reviewing each individual accident.

5.4 DOCUMENTATION

Accident investigations will be documented using photographs and by sequestering faulty tools and equipment that were involved, as well as by completing the appropriate forms (see Section 6.0, Record Keeping).

6.0 RECORD KEEPING

An Accident Investigation Interview Report (Attachment 1), Guide for Identifying Causal Factors and Corrective Actions (Attachment 2), and Corrective Action Program Schedule (Attachment 3) will be completed for each work-related incident, injury, or illness that occurs.

7.0 REFERENCES

Fed-OSHA. 1997. 29 CFR 1904, *Recording and Reporting Occupational Injuries and Illnesses.*

Cal-OSHA. 1997. 8 CCR 3203, *Injury and Illness Prevention Program.*

National Safety Council. 1997. Accident Prevention Manual for Business and Industry: Administration and Programs. 10th ed.

8.0 ATTACHMENTS

1. Accident Investigation Interview Report
2. Guide for Identifying Causal Factors and Corrective Actions
3. Corrective Action Program Schedule

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ACCIDENT INVESTIGATION INTERVIEW REPORT

Name of Interviewer: ______________________ Name of Interviewee: ______________________

Department of Interviewer: ______________________ Department of Interviewee: ______________________

Project: ______________________ Time shift began: ____________ a.m/p.m.

Project activity during this shift: ______________________

Accident date: ______________________ Time of accident: ____________ a.m/p.m.

Date accident was reported? ______________________

Any injuries involved? Yes ☐ No ☐ ______________________

Location of accident (include address, city, county, zip code): ______________________

____

Name of witnesses: ______________________

____

____

____

What was/were employee(s) doing when accident occurred? Be specific (i.e., walking, lifting, operating machinery, etc.)?

____

____

____

Please describe fully the events that resulted in the accident. Tell what happened and how it happened. (Do not describe the nature of the injury or property damage.).

____

____

____

What machine, tool, or object was most closely connected to the accident? (e.g, machine employee struck against or which struck him, vehicle employee was driving)?

____

____

____

Nature of injury or property damage.

____

____

____

Additional Information or drawings can be included on the reverse side of this form.

Forward completed form to the appropriate HSM, office HSC, SHSC, and CHSD.

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ATTACHMENT 2

GUIDE FOR IDENTIFYING CAUSAL FACTORS AND CORRECTIVE ACTIONS

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GUIDE FOR IDENTIFYING CAUSAL FACTORS and CORRECTIVE ACTIONS

Project/Department: ______________________ Investigators: ______________________

Accident Date/Time: ______________________ Date/Time of Investigation: ______________________

PART 1 - EQUIPMENT

Answer questions by placing an *x* in the appropriate box or circle (Y=Yes, N=No)

Y N 1.0 **WAS A HAZARDOUS CONDITION(S) A CONTRIBUTING FACTOR?**

Y N	CAUSAL FACTORS	COMMENTS	POSSIBLE CORRECTIVE ACTIONS	RECOMMENDED CORRECTIVE ACTIONS	TARGET COMPLETION DATE
Y N	1.1 Did any defect(s) in equipment/tool(s)/material contribute to hazardous condition(s)?		Review procedure for inspecting, reporting, maintaining, repairing, replacing, or recalling defective equipment/tool(s)/material used.		
Y N	1.2 Was the hazardous condition(s) recognized? If yes, answer A and B. If no, proceed to 1.3.		Perform job hazard analysis. Improve employee ability to recognize existing or potential hazardous conditions. Provide test equipment, as required, to detect hazard. Review any change or modification of equipment/tool(s)/material.		
Y N	A. Was the hazardous condition(s) reported?		Train employees in reporting procedures. Stress individual acceptance of responsibility.		
Y N	B. Was employee(s) informed of the hazardous condition(s) and the job procedures for dealing with it as an interim measure?		Review job procedures for hazard avoidance. Review supervisory responsibility. Improve supervisor-employee communications. Take action to remove or minimize hazard.		
Y N	1.3 Was there an equipment inspection procedure(s) to detect the hazardous condition(s)?		Develop and adopt procedures (for example, an inspection system) to detect hazardous conditions. Conduct test.		
Y N	1.4 Did the existing equipment inspection procedure(s) detect the hazardous condition(s)?		Review procedures. Change frequency or comprehensiveness. Provide test equipment as required. Improve employee ability to detect defects and hazardous conditions. Change job procedures as required.		
Y N	1.5 Was the correct equipment/tool(s)/material used?		Specify correct equipment/tool(s)/material in job procedures.		
Y N	1.6 Was the correct equipment/tool(s)/material readily available?		Provide correct equipment/tool(s)/material. Review purchasing specifications and procedures. Anticipate future requirements.		
Y N	1.7 Did employee(s) know where to obtain equipment/tool(s)/material required for the job?		Review procedures for storage, access, delivery, or distribution. Review job procedures for obtaining equipment/tool(s)/material.		
Y N	1.8 Was substitute equipment/tool(s)/material used in place of correct one?		Provide correct equipment/tool(s)/material. Warn against use of substitutes in job procedures and in job instruction.		
Y N	1.9 Did the design of the equipment/tool(s) create operator stress or encourage operator error?		Review human factors engineering principles. Alter equipment/tool(s) to make it more compatible with human capability and limitations. Review purchasing procedures and specifications. Check out new equipment and job procedures involving new equipment before putting into service. Encourage employees to report potential hazardous conditions created by equipment design.		
Y N	1.10 Did the general design or quality of the equipment/tool(s) contribute to a hazardous condition?		Review criteria in codes, standards, specifications, and regulations. Establish new criteria as required.		
○	1.11 List other causal factors in "Comment" column.				

SOURCE: National Safety Council, Accident Prevention Manual for Business and Industry: Administration and Programs, 1988

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PART 2 - ENVIRONMENTAL

Answer questions by placing an x in the appropriate box or circle (Y=Yes, N=No)

Y	N		Causal Factors	Comments	Possible Corrective Actions	Recommended Corrective Actions	Target Completion Date
○	☐	2.0	**WAS THE LOCATION/POSITION OF EQUIPMENT/MATERIALS/EMPLOYEE(S) A CONTRIBUTING FACTOR ?**				
○	☐	2.1	Did the location/position of equipment/material/employee(s) contribute to a hazardous condition?		Perform job hazard analysis. Review job procedures. Change the location, position, or layout of the equipment. Change position of employee(s). Provide guardrails, barricades, barriers, warning lights, signs, or signals.		
☐	○	2.2	Was the hazardous condition(s) recognized? If yes, answer A and B. If no, proceed to 2.3.		Perform job hazard analysis. Improve employee ability to recognize existing or potential hazardous conditions. Provide test equipment, as required, to detect hazard. Review any change or modification of equipment/tool(s)/ material.		
☐	○		A. Was the hazardous condition(s) reported?		Train employees in reporting procedures. Stress individual acceptance of responsibility.		
☐	○		B. Was employee(s) informed of the job procedure for dealing with the hazardous condition as an interim action?		Review job procedures for hazard avoidance. Review supervisory responsibility. Improve supervisor-employee communications. Take action to remove or minimize hazard.		
☐	○	2.3	Was employee(s) supposed to be in the vicinity of the equipment/material?		Review job procedures and instruction. Provide guardrails, barricades, barriers, warning lights, signs, or signals.		
☐	○	2.4	Was the hazardous condition created by the location/position of equipment/material visible to employee(s)?		Change lighting or layout to increase visibility of equipment. Provide guard-rails, barricades, barriers, warning lights, signs, or signals, floor stripes, etc.		
☐	○	2.5	Was there sufficient workspace?		Review workspace requirements and modify as required.		
○	☐	2.6	Were environmental conditions a contributing factor (for example, illumination, noise levels, air contaminant, temperature extremes, ventilation, vibration, radiation)?		Monitor, or periodically check environmental conditions as required. Check results against acceptable levels. Initiate action for those found unacceptable.		
○		2.7	List other causal factors in "Comment" column.				

PART 3 - PEOPLE

Y	N		Causal Factors	Comments	Possible Corrective Actions	Recommended Corrective Actions	Target Completion Date
○	☐	3.0	**WAS THE JOB PROCEDURE(S) USED A CONTRIBUTING FACTOR?**				
☐	○	3.1	Was there a written or known procedure (rules) for this job ?		Perform job hazard analysis and develop safe job procedures.		
☐	○		If yes, answer A, B, and C. If no, proceed to 3.2.				
☐	○		A. Did job procedures anticipate the factors that contributed to the accident?		Perform job hazard analysis and change job procedures.		
☐	○		B. Did employee(s) know the job procedure?		Improve job instruction. Train employees in correct job procedures.		
○	☐		C. Did employee(s) deviate from the known job procedure?		Determine why. Encourage all employees to report problems with an established procedure to supervision. Review job procedure and modify if necessary. Counsel or discipline employee. Provide closer supervision.		
☐	○	3.2	Was employee(s) mentally and physically capable of performing the job?		Review employee requirements for the job. Improve employee selection. Remove or transfer employees who are temporarily, either mentally or physically, incapable of performing the job.		
○	☐	3.3	Were any tasks in the job procedure too difficult to perform (for example, excessive concentration or physical demands)?		Change job design and procedures.		
○	☐	3.4	Is the job structured to encourage or require deviation from job procedures (for example, incentive, piecework, work pace)?		Change job design and procedures.		
○		3.5	List other causal factors in "Comment" column.				
○	☐	3.6	**WAS LACK OF PERSONAL PROTECTIVE EQUIPMENT OR EMERGENCY EQUIPMENT A CONTRIBUTING FACTOR IN THE INJURY?**				

SOURCE: National Safety Council, Accident Prevention Manual for Business and Industry: Administration and Programs, 1988

PART 3 - PEOPLE (Continued)

Answer questions by placing an x in the appropriate box or circle (Y=Yes, N=No)

NOTE: THE FOLLOWING CAUSAL FACTORS RELATE TO THE INJURY.

Y/N	CAUSAL FACTORS	COMMENTS	POSSIBLE CORRECTIVE ACTIONS	RECOMMENDED CORRECTIVE ACTIONS	TARGET COMPLETION DATE
☐ Y ○ N	3.7 Was appropriate personal protective equipment (PPE) specified for the task or job?		Review methods to specify PPE requirements.		
	If yes, answer A, B, and C. If no, proceed to 3.8.				
☐ Y ○ N	A. Was appropriate PPE available?		Provide appropriate PPE. Review purchasing and distribution procedures.		
☐ Y ○ N	B. Did employee(s) know that wearing specified PPE was required?		Review job procedures. Improve job instruction.		
☐ Y ○ N	C. Did employee(s) know how to use and maintain the PPE?		Improve job instruction.		
☐ Y ○ N	3.8 Was the PPE used properly when the injury occurred?		Determine why and take appropriate action. Implement procedures to monitor and enforce use of PPE.		
☐ Y ○ N	3.9 Was the PPE adequate?		Review PPE requirements. Check standards, specification, and certification of the PPE.		
☐ Y ○ N	3.10 Was emergency equipment specified for this job (for example, emergency showers, eyewash fountains)?		Provide emergency equipment as required.		
	If yes, answer the following. If no, proceed to Part 4.				
☐ Y ○ N	A. Was emergency equipment readily available?		Install emergency equipment at appropriate locations.		
☐ Y ○ N	B. Was emergency equipment properly used?		Incorporate use of emergency equipment in job procedures.		
☐ Y ○ N	C. Did emergency equipment function properly?		Establish inspection/monitoring system for emergency equipment. Provide for immediate repair of defects.		
○	3.11 List other causal factors in "Comment" column.				

PART 4 - MANAGEMENT

○ Y ☐ N 4.0 WAS A MANAGEMENT SYSTEM DEFECT A CONTRIBUTING FACTOR?

Y/N	CAUSAL FACTORS	COMMENTS	POSSIBLE CORRECTIVE ACTIONS	RECOMMENDED CORRECTIVE ACTIONS	TARGET COMPLETION DATE
○ Y ☐ N	4.1 Was there a failure by supervision to detect, anticipate, or report a hazardous condition?		Improve supervisor capability in hazard recognition and reporting procedures.		
○ Y ☐ N	4.2 Was there a failure by supervision to detect or correct deviations from job procedure?		Review job hazard analysis and job procedures. Increase supervisor monitoring. Correct deviations.		
☐ Y ○ N	4.3 Was there a supervisor/employee review of hazards and job procedures for tasks performed infrequently? (Not applicable to all accidents.)		Establish a procedure that requires a review of hazards and job procedures (preventive actions) for tasks performed infrequently.		
☐ Y ○ N	4.4 Was supervisor responsibility and accountability defined and understood?		Define and communicate supervisor responsibility and accountability. Test for understandability and acceptance.		
☐ Y ○ N	4.5 Was supervisor adequately trained to fulfill assigned responsibility in accident prevention?		Train supervisors in accident prevention fundamentals.		
○ Y ☐ N	4.6 Was there a failure to initiate corrective action for a known hazardous condition that contributed to this accident?		Review management safety policy and level of risk acceptance. Establish priorities based on potential severity and probability of recurrence. Review procedure and responsibility to initiate and carry out corrective actions. Monitor progress.		
○	4.7 List other causal factors in "Comment" column.				

(x) ITEM IS A CAUSAL FACTOR

[x] ITEM IS NOT A CAUSAL FACTOR

SOURCE: National Safety Council, Accident Prevention Manual for Business and Industry: Administration and Programs, 1988

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ATTACHMENT 3

CORRECTIVE ACTION PROGRAM SCHEDULE

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ATTACHMENT 1

ACCIDENT INVESTIGATION INTERVIEW REPORT

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CORRECTIVE ACTION PROGRAM SCHEDULE

LOCATION:

ATTENTION:

DATE:

ITEM NO	DESCRIPTION OF ITEM	ASSIGNED TO	DATE ASSIGNED	COMPLETION TARGET	DATE COMPLETED	STATUS

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ATTACHMENT C

MATERIAL SAFETY DATA SHEETS

MSDSs

SYSTEM.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 1 - Slight
Flammability Rating: 4 - Extreme (Flammable)
Reactivity Rating: 2 - Moderate
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. Higher concentrations can produce central nervous system depression, narcosis, and unconsciousness.
Ingestion:
Swallowing small amounts is not likely to produce harmful effects. Ingestion of larger amounts may produce abdominal pain, nausea and vomiting. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms are expected to parallel inhalation.
Skin Contact:
Irritating due to defatting action on skin. Causes redness, pain, drying and cracking of the skin.
Eye Contact:
Vapors are irritating to the eyes. Splashes may cause severe irritation, with stinging, tearing, redness and pain.
Chronic Exposure:
Prolonged or repeated skin contact may produce severe irritation or dermatitis.
Aggravation of Pre-existing Conditions:
Use of alcoholic beverages enhances toxic effects. Exposure may increase the toxic potential of chlorinated hydrocarbons, such as chloroform, trichloroethane.

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing

is difficult, give oxygen. Get medical attention.
Ingestion:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately.
Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting upper and lower eyelids occasionally. Get medical attention.

5. Fire Fighting Measures

Fire:
Flash point: -20C (-4F) CC
Autoignition temperature: 465C (869F)
Flammable limits in air % by volume:
lel: 2.5; uel: 12.8
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Vapors can flow along surfaces to distant ignition source and flash back. Contact with strong oxidizers may cause fire. Sealed containers may rupture when heated. This material may produce a floating fire hazard. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, alcohol foam or carbon dioxide. Water may be ineffective. Water spray may be used to keep fire exposed containers cool, dilute spills to nonflammable mixtures, protect personnel attempting to stop leak and disperse vapors.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB(R) solvent adsorbent is recommended for spills of this product.

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Acetone:
-OSHA Permissible Exposure Limit (PEL):
1000 ppm (TWA)

-ACGIH Threshold Limit Value (TLV):
500 ppm (TWA), 750 ppm (STEL) A4 - not classifiable as a human carcinogen
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, a half-face organic vapor respirator may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece organic vapor respirator

may be worn up to 50 times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-face piece positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Clear, colorless, volatile liquid.
Odor:
Fragrant, mint-like
Solubility:
Miscible in all proportions in water.
Specific Gravity:
0.79 @ 20C/4C
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
56.5C (133F) @ 760 mm Hg
Melting Point:
-95C (-139F)
Vapor Density (Air=1):
2.0
Vapor Pressure (mm Hg):
400 @ 39.5C (104F)
Evaporation Rate (BuAc=1):
ca. 7.7

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
Carbon dioxide and carbon monoxide may form when heated to

decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Concentrated nitric and sulfuric acid mixtures, oxidizing materials, chloroform, alkalis, chlorine compounds, acids, potassium t-butoxide.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

11. Toxicological Information

Oral rat LD50: 5800 mg/kg; Inhalation rat LC50: 50,100mg/m3; Irritation eye rabbit, Standard Draize, 20 mg severe; investigated as a tumorigen, mutagen, reproductive effector.

--------\Cancer Lists\--

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC
Acetone (67-64-1)	No	No	N

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material is expected to readily biodegrade. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material may be moderately degraded by photolysis. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 350LB

International (Water, I.M.O.)

Proper Shipping Name: ACETONE
Hazard Class: 3.1
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 350LB

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\-----------------------

Ingredient	TSCA	EC	Japan
Acetone (67-64-1)	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\-----------------------

Ingredient	Korea	--Canada-- DSL	NDSL
Acetone (67-64-1)	Yes	Yes	No

--------\Federal, State & International Regulations - Part 1\------

Ingredient	-SARA 302- RQ	TPQ	------SARA List	Chem
Acetone (67-64-1)	No	No	Yes	

--------\Federal, State & International Regulations - Part 2\------

Ingredient	CERCLA	-RCRA- 261.33	-TS 8(

Acetone (67-64-1)	5000	U002	No

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Chemical Weapons Convention:  No     TSCA 12(b):  Yes    CDTA:  Yes
SARA 311/312:  Acute: Yes      Chronic: No   Fire: Yes Pressure: No
Reactivity: No          (Pure / Liquid)
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Australian Hazchem Code: 2[Y]E
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.
Label First Aid:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 8.
Disclaimer:
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Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

Please reduce your browser font size for better viewing and printing.

MSDS *Material Safety Data Sheet*

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

MALLINCKRODT   J.T.Baker

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

NITRIC ACID 1.0 N AND 2.0 N VOLUMETRIC SOLUTIONS

MSDS Number: N3659 --- *Effective Date: 10/15/99*

1. Product Identification

Synonyms: Azotic acid solution; nitric acid 6.3%; nitric acid 1.0 N volumetric solution; nitric acid 2.0 N volumetric solution; nitric acid 12.6%
CAS No.: 7697-37-2
Molecular Weight: 63.00
Chemical Formula: HNO3 in H2O
Product Codes:
J.T. Baker: 5639
Mallinckrodt: 3510

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent
Nitric Acid	7697-37-2	6 - 13%
Water	7732-18-5	> 87%

3. Hazards Identification

Emergency Overview

POISON! DANGER! OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL

IF SWALLOWED. HARMFUL IF INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Oxidizer)
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Yellow (Reactive)

Potential Health Effects

Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison.

Inhalation:
Corrosive! Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract.
Ingestion:
Corrosive! Swallowing nitric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract.
Skin Contact:
Corrosive! Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color.
Eye Contact:
Corrosive! Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Chronic Exposure:
Long-term exposure to concentrated vapors may cause erosion of teeth and lung damage. Long-term exposures seldom occur due to the corrosive properties of the acid.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders, eye disease, or cardiopulmonary diseases may be more susceptible to the effects of this substance.

4. First Aid Measures

Immediate first aid treatment reduces the health effects of this substance.
Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

5. Fire Fighting Measures

Fire:
Not combustible, but substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition. Can react with metals to release flammable hydrogen gas.
Explosion:
May react explosively with combustible organic or readily oxidizable materials such as: alcohols, turpentine, charcoal, organic refuse, metal powder, hydrogen sulfide, etc.
Fire Extinguishing Media:
If involved in a fire, use water spray.
Special Information:
Increases the flammability of combustible, organic and readily oxidizable materials. In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash, lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker NEUTRASORB(R) or TEAM(R) 'Low Na+' acid neutralizers are recommended for spills of this product.

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect from physical damage and direct sunlight. Isolate from incompatible substances. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
For Nitric Acid:
OSHA Permissible Exposure Limit (PEL):
2 ppm (TWA)
ACGIH Threshold Limit Value (TLV):
2 ppm (TWA); 4 ppm (STEL)
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Nitric acid is an oxidizer and should not come in contact with cartridges and canisters that contain oxidizable materials, such as activated charcoal. Canister-type respirators using sorbents are ineffective.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Colorless to yellowish liquid.
Odor:
Suffocating, acrid.
Solubility:
Infinitely soluble.
Specific Gravity:
No information found.
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100 (as water and acid)
Boiling Point:
No information found.
Melting Point:
No information found.
Vapor Density (Air=1):
No information found.
Vapor Pressure (mm Hg):
No information found.
Evaporation Rate (BuAc=1):
No information found.

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Containers may burst when heated.
Hazardous Decomposition Products:
When heated to decomposition, emits toxic nitrogen oxides fumes and hydrogen nitrate.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
A dangerously powerful oxidizing agent, concentrated nitric acid is incompatible with most substances, especially strong bases, metallic powders, carbides, hydrogen sulfide, turpentine, and combustible organics.
Conditions to Avoid:
Heat and incompatibles.

11. Toxicological Information

For Nitric Acid: Investigated as a mutagen and reproductive effector.

--------\Cancer Lists\--

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC
Nitric Acid (7697-37-2)	No	No	N
Water (7732-18-5)	No	No	N

12. Ecological Information

Environmental Fate:
No information found.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste facility. Although not a listed RCRA hazardous waste, this material may exhibit one or more characteristics of a hazardous waste and require appropriate analysis to determine specific disposal requirements. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)
Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 20L

International (Water, I.M.O.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)

Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 20L

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\-----------------------

Ingredient	TSCA	EC	Japan
Nitric Acid (7697-37-2)	Yes	Yes	Yes
Water (7732-18-5)	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\-----------------------

		--Canada--	
Ingredient	Korea	DSL	NDSL
Nitric Acid (7697-37-2)	Yes	Yes	No
Water (7732-18-5)	Yes	Yes	No

--------\Federal, State & International Regulations - Part 1\------

	-SARA 302-		------SARA	
Ingredient	RQ	TPQ	List	Chem
Nitric Acid (7697-37-2)	1000	1000	Yes	
Water (7732-18-5)	No	No	No	

--------\Federal, State & International Regulations - Part 2\------

		-RCRA-	-TS
Ingredient	CERCLA	261.33	8(
Nitric Acid (7697-37-2)	1000	No	No
Water (7732-18-5)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: Yes (Mixture / Liquid)

Australian Hazchem Code: 2PE
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: **3** Flammability: **0** Reactivity: **0** Other: **Oxidizer**
Label Hazard Warning:
POISON! DANGER! OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED. HARMFUL IF INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.
Label Precautions:
Do not get in eyes, on skin, or on clothing.
Do not breathe vapor or mist.
Use only with adequate ventilation.
Wash thoroughly after handling.
Keep from contact with clothing and other combustible materials.
Store in a tightly closed container.
Label First Aid:
In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases call a physician.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 3.
Disclaimer:
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Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

ATTACHMENT D

EQUIPMENT SOPs/CALIBRATION LOG

EXIT Key - Cancels the key with no more changes

GRAPH Key - Prints graph of the recorded data

Keypad - Used to set up and calibrate the MicroTIP, and allows the user to manipulate the concentrations measured and recorded in various ways. The MicroTIP has 16 keys for direct numeric entry and for using the instrument's functions.

LIGHT Key - Shows intensity of detector lamp

MAX Key - Displays highest concentration measured

PLAY Key - Replays recorded data on the display

PRINT Key - Prints table of recorded data

SETUP Key - Sets data time and options for keys

Span Gas - Gas that will be used to calibrate the MicroTIP. The span gas contains a known concentration of a photoionizable gas or vapor and is used to set the sensitivity. Usually isobutylene at 100 parts per million (ppm) in air is recommended as span gas.

TUTOR Key - Begins tutorial session (While in the tutorial, keypresses have no effect on MicroTIP's operation. Press the EXIT key to end the session.)

Zero Air - Contains no ionizable gases or vapors and is used to set the zero point of the MicroTIP during calibration. Usually clean ambient air will be suitable as zero air. However, if there is any doubt as to the quality of the ambient air, then a commercial source of zero grade air and a sampling bag must be used.

4.0 RESPONSIBILITIES

4.1 Health and Safety Manager (HSM)

The HSM is responsible for approving instrument procedures for issue and general implementation of this procedure.

4.2 Health and Safety Coordinator (HSC)

The HSC arranges for the training of personnel in the use of the instruments covered by the procedures.

4.3 Site Health and Safety Coordinator (SHSC)

The SHSC is responsible for general field implementation of the procedures, which includes instrument calibration, operation, maintenance, and recordkeeping.

5.0 PROCEDURES

5.1 Check Battery Voltage

Before any instrument is used by field personnel, the battery voltage will be checked.

Turn on the MicroTIP by pressing the back of the power switch. The pump will start and the instrument will need to warm up (less than three minutes). Press the BATT key. The normal operating voltage is between 6 and 8.5 volts.

5.2 Calibration

5.2.1 Parts Required

- Calibration kit, part no. 390033, consisting of:
  - Gas pressure regulator with contents gauge, to fit a 6D size cylinder of span gas
  - Gas sampling bag
  - Adapter tubing with fittings for regulator and MicroTIP inlet (attached to gas sampling bag)
- Span gas cylinder of isobutylene at 100 parts per million (ppm) ±5% in air.

5.2.2 Steps Required

The instrument should be calibrated at least once a day. Zero air is used to set the zero point. Clean, ambient air is suitable as zero air. Span gas is used to set the sensitivity.

Isobutylene at 100 ppm in air is recommended as span gas. To calibrate the instrument use the calibration kit as follows:

1. Connect the supplied regulator to the span gas cylinder (typically 100 ppm isobutylene). Hand tighten the fittings.

2. Open the valve on the gas bag by turning the valve stem fully counterclockwise.

3. Attach the nut to the regulator. Hand tighten the fittings.

4. Turn the regulator knob counterclockwise about half a turn to start the flow of gas.

5. Fill the gas bag about half full and then close the regulator. Turn the valve stem fully clockwise to stop the flow of gas.

6. Disconnect the bag from the adapter and press gently on the bag to empty it. Flush the bag a few more times with the span gas and then fill it with the span gas.

7. Close the gas bag by turning the valve clockwise.

8. Press SETUP and select the desired Cal Memory (based on which span gas is used) with the arrow keys and press ENTER. Press EXIT to leave setup.

9. Press CAL and enter the desired response factor (refer to the user's manual section on response factors for specific response factors of gases and vapors). If a specific compound is not being looked for, then enter 1.00. Section 5.3.3 of this SOP explains interpretation of results using the response factor.

10. Expose MicroTIP to zero air (clean, ambient air is suitable). Press ENTER and the instrument sets its zero point.

11. MicroTIP then asks for the span gas concentration. Enter the known span gas concentration and then connect the span gas bag adapter to the inlet.

12. Press ENTER and the instrument sets its sensitivity.

13. When the display reverts to normal, the instrument is calibrated and ready for use. Remove the span gas bag from the inlet.

14. Record readings on the Instrument Calibration Log (Attachment 2).

The MicroTIP has 10 Cal Memories and can be calibrated with 10 different span gases or response factors if desired. Only one Cal Memory can be used at a time. Each stores a different response factor, zero point, and sensitivity. To program:

1. Press SETUP and select the desired Cal memory (1-10) with the arrow keys.
2. Exit from SETUP and press the CAL key.
3. Enter the desired response factor and press ENTER.
4. Follow the displayed calibration instructions.

5.3 MicroTIP HL-2000 Operation

Check to see that the MicroTIP HL-2000 User's Manual is in the instrument case before taking the monitor in the field or shipping the device to the field.

The MicroTIP layout and normal display are shown in Attachment 1. Turn the instrument on by pressing the back of the power switch (see Attachment 1). The pump will start and the message "Warming up now, please wait" will be displayed. Within three minutes the following information will appear as part of the normal display (see Attachment 1): instrument status, current detected concentration, event number (if the datalogger is on), time, and date.

The MicroTIP operates automatically. The user reads the concentration directly from MicroTIP's display which updates itself each half second.

The minimum, maximum, and average concentrations measured are measured over 15-second periods and are automatically recorded in MicroTIP's datalogging memory if the datalogger is turned on. MicroTIP's memory holds 12 hours of concentration data.

All information entered from the keypad and stored in the MicroTIP memory is retained when the instrument is switched off. The clock and calendar continue to operate.

5.3.1 Specific Functions

The following is a description of each key function:

DISPLAY - If a numerical display is shown, pressing the DISPLAY key will change it to a bar graph. If the bar graph is shown, pressing the DISPLAY key changes it to a numerical display. The bar graph range is selected with the SETUP key.

LIGHT - LIGHT switches the backlighting between high and normal intensity and will turn backlighting off. The current intensity of the detector ultraviolet (UV) lamp is also displayed when the key is pressed. The brighter backlighting consumes more power and is recommended for use only in very dark locations.

BATT - Pressing the BATT key displays the current battery level. When "LoBat" is displayed there is approximately 10 minutes of operation left. To continue normal operation, replace the discharged battery pack with a fully charged one.

MAX - Maximum concentration is shown when the MAX key is pressed. The maximum concentration, event during which it was encountered, time, and date of the occurrence will be displayed for 15 seconds. By pressing the MAX key and then pressing the CLEAR key twice, the max register will be cleared.

CLEAR - If a number is entered in error, press CLEAR to erase the entry and re-enter the correct number.

EVENT - The EVENT key may be used to identify a particular sample or sampling location in memory. Recorded data may be displayed, printed, and removed from the datalogger by specifying a start and stop event number (refer to the MicroTIP User's Manual for complete instructions and limitations).

EXIT - EXIT terminates all MicroTIP functions except DISPLAY. When this key is pressed the display reverts to normal. Most functions exit automatically if no key is pressed for 15 seconds.

SETUP - SETUP allows the instrument to be set up for a specific application. The current date and time are also set with this key.

To set up the instrument:

1. Press the SETUP key.

2. The first option sets the full scale range for the bar graph display, graph output, audio output, and the 1 volt analog output. Use the up and down arrow keys to select the 20, 200, or 2000 ppm range and press ENTER. This does not affect the instrument's ability to read up to 2500 ppm.

3. Select Cal Memory 1 with the up and down arrow keys and press ENTER.

4. Enter the correct values for the current time, pressing ENTER after each value.

5. Enter the numerical values for the day, month, and year, pressing ENTER after each selection.

TUTOR - Pressing this key begins a tutorial session which displays a two-line description of the function of each key. Press each key and read the tutorial display. Pressing EXIT ends the tutorial session. While in the tutorial session, key presses have no effect on MicroTIP's operation.

AUDIO - To connect the headset (part no. 395030), remove the dust cap from the I/O connector and plug in the headset. Press the AUDIO key and use the arrow keys to select one of three options for audio output and press ENTER.

ALARM - Pressing ALARM will display the current alarm level and allows a new alarm level to be entered. If the alarm value is correct, press EXIT to return to the normal display. If a new value is to be set, enter the value and press ENTER.

PLAY - This key plays back previously recorded data.

1. Press PLAY and two options will be available. Pressing the ENTER key begins playback where it was last stopped. Press the SETUP (*) key to set the playback options.

2. Enter the start event.

3. Select which value is to be displayed.

4. The data can be played back in either numerical or graphical display by pressing the DISPLAY key.

When the instrument is playing back recorded data it is also measuring and recording real-time concentrations even though the instrument status is "play." Press EXIT to return to the normal display.

PRINT and GRAPH - PRINT key allows the data to be printed while the MicroTIP is measuring real-time concentrations. When used with a compatible printer, the GRAPH key prints the recorded data in graphical form. Refer to the MicroTIP User's Manual for complete instructions for these operations.

5.3.2 Detector Lamp Selection

The MicroTIP is supplied with a UV lamp that produces an energy of 10.6 electron-volts (eV). With this standard lamp installed, MicroTIP responds well to gases and vapors that ionize at 10.6 eV or less. If necessary, the MicroTIP may be equipped with lamps that produce different amounts of energy. The lamp selected must have an energy (in eV) greater than the ionization potential of the analyte it is detecting.

5.3.3 Interpreting the Results

The MicroTIP will indicate a 1:1 response ratio if exposed to the same gas as the gas to which it was calibrated. However, if the MicroTIP is exposed to a gas or vapor (or mixture of gases/vapors) that are not the same as the calibration gas, it will not detect at a 1:1 response ratio. When the MicroTIP has been calibrated to the span gas, the response factor is:

$$\frac{\text{known concentration of target gas}}{\text{displayed concentration of target gas (as a ppm} - \text{equivalent)}}$$

A response factor may be used to relate a particular gas to the calibration gas (such as methane as compared to isobutylene as the calibration gas). Response factors can be input when calibrating the MicroTIP (see Section 5.2.2, Step 9). When computing the concentration, the MicroTIP multiplies the detected concentration by the response factor and displays the result. If the response factor is 1.0 then the concentration is not changed. The instruction manual provides response factors for common gases that may be encountered.

If the MicroTIP response factor is set at 1.0 when using the instrument to detect gases or vapors other than the span gas, then the person reading the display should manually perform the following computation:

Actual airborne concentration of target gas =
displayed concentration x response factor of target gas

Record results on the Sir Air Surveillance Record (Attachment 3).

5.4 High Sensitivity Operation

The MicroTIP can be used as a high sensitivity leak detector for photoionizable chemicals. In High Sensitivity operation, MicroTIP does not read directly in ppm but displays a reading proportional to the concentration of photoionizable gases and vapors detected.

During high sensitivity calibration, no span gas is required. MicroTIP zeros its reading with zero air and then sets itself to the maximum sensitivity.

1. Press SETUP. Select the 0-20 ppm display range with the arrow keys and press ENTER.

2. Select High Sensitivity with the arrow keys and press ENTER.

3. Press EXIT. Select the bar graph with the DISPLAY key.

4. Press CAL and calibrate the MicroTIP with zero air. High Sensitivity operation does not require a span gas.

5. Press AUDIO and select "continuous audio" with the arrow keys.

5.5 Warning Limitations

The MicroTIP does not distinguish between individual pollutants. The reading displayed represents the total concentration of all photoionizable chemicals present in the sample.

This device is not intended for constant use with flammable concentrations of gases.

Calibration, maintenance, and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.

Exercise care in handling battery packs in order not to short the terminals with conducting materials such as rings, bracelets, and keys. The battery or conductor may overheat and cause burns.

5.6 Accessories and Other Devices

The MicroTIP User's Manual contains detailed instructions for the following:

A. Printer
B. Computer
C. Chart recorder
D. Sample bag
E. 3-meter sample line (for remote sampling)
F. Shoulder strap
G. Headset
H. Replacement detector lamps

6.0 MAINTENANCE

6.1 Charging The Battery

When the instrument status reads "LoBat," the MicroTIP battery pack requires recharging. A fully charged battery powers the instrument for 7 hours. If the instrument is to be used for more than 7 hours, carry a spare battery pack. Approximately 8 hours is required to recharge a fully discharged battery pack (NOTE: use only the HL-2000 battery charger).

If the MicroTIP is left for more than three days without a charged battery, recorded data and setup parameters will be lost. To avoid loss of data, charge the battery pack for at least 8 hours and attach it back to the instrument.

Note: If MicroTIP is not used regularly, the battery should be charged at least once a month for between 8 and 72 hours.

6.2 Cleaning the Lamp Window

During the course of normal operation a film builds up on the window of the detector lamp. The window should be cleaned every 24 hours of operation using the following procedure:

1. Turn off the instrument.

2. Hold the black detector housing in one hand and unscrew it from the body of the instrument. Remove the housing, being careful not to lose the O-ring seal on top to the photoionization detector. The detector cell, lamp holder, and high frequency (HF) driver circuit board are now exposed.

3. Unplug the red and yellow wires from the HF driver circuit board.

4. Locate the black ground wire. Loosen the screw on the HF driver circuit board and disconnect the black wire.

5. Hold the lamp holder in one hand so it will not rotate and carefully unscrew the detector cell with the red, yellow, and black wires attached. (NOTE: Do not touch the fine wire mesh inside the detector cell).

6. Leaving the lamp spring in place, remove the lamp from the lamp holder.

7. To remove the film, gently rub the window of the lamp with a lint-free tissue moistened with HPLC or spectroscopic grade methanol.

8. Allow the window to dry. Then, using a lint-free tissue (touching the lamp as little as possible), replace it in the lamp holder.

9. Replace the detector cell squarely on the lamp holder. Finger tighten the detector cell, being careful not to overtighten.

10. Replace the black wire below the screw on the HF driver circuit board and tighten the screw down. Plug the yellow wire into the gold pin and the red wire into the silver pin on the HF driver circuit board.

11. Check the lamp holder and ensure it is securely seated by hand. Check that the O-ring seal is in position.

12. Replace the detector housing and tighten by hand.

6.3 Additional Maintenance

The MicroTIP User's Manual contains detailed instructions for the following:

A. replacing the detector UV lamp
B. replacing the inlet filter
C. replacing the sample pump

7.0 RECORDS

7.1 Procedure

A copy of each approved procedure and revision shall be retained as a permanent record in accordance with company procedures. Monitoring results will be documented on the Site Air Surveillance Record (Attachment 3).

7.2 Calibration

The Instrument Calibration Log (Attachment 2) will be maintained in the Health and Safety portion of the project files and/or on a log with the instrument.

8.0 SHIPPING FEDERAL EXPRESS

The MicroTIP does not contain any hazardous materials or components. However, some of the calibration gases used for calibrating the instrument are classified as hazardous materials.

Any person involved with the handling or transportation of hazardous materials or who has the potential to be exposed to hazardous materials must receive Department of Transportation (DOT) HM-126F training. Shipping labels must comply with DOT regulations. The following information will need to be listed on both the package being shipped and the paperwork accompanying the shipment (from 49 CFR Hazardous Materials, Table 172.101):

- proper shipping name
- hazard class
- identification number
- packing group
- labels required
- special provisions
- packaging authorization
- quantity limitations by air

9.0 REFERENCES

Photovac. 1992. MicroTIP HL-2000 User's Manual.

10.0 ATTACHMENTS

1. MicroTIP Layout and Normal Display
2. Instrument Calibration Log
3. Site Air Surveillance Record

ATTACHMENT 1

MICROTIP LAYOUT AND NORMAL DISPLAY

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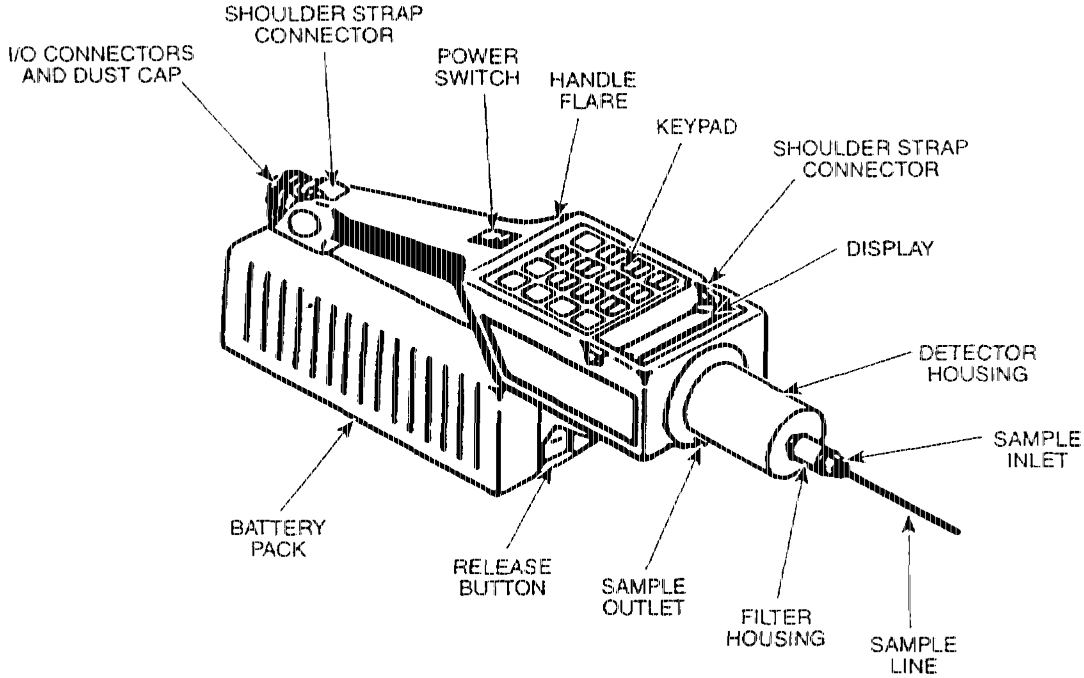
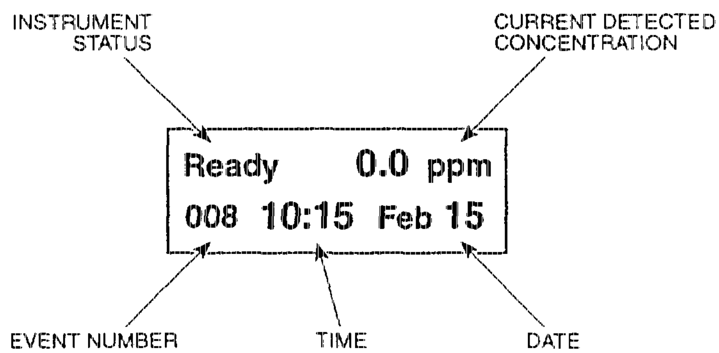


A. MicroTIP Layout



B. Normal Display

SOURCE: Photovac, "MicroTIP HL-2000 User's Manual"

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MicroTIP Layout and Normal Display

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ATTACHMENT 2

INSTRUMENT CALIBRATION LOG

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OGDEN

INSTRUMENT CALIBRATION LOG

Project: ______________________

JOB #_ ______________________

Location: ______________________

Date/Time	Instrument	Serial Number	Span Gas Substance	Span Conc.	Instrument Reading at Calibration	Instrument Calibration Reading Post Sampling	Comments

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ATTACHMENT 3

SITE AIR SURVEILLANCE RECORD

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SITE AIR SURVEILLANCE RECORD

SITE INFORMATION		Page ____ of ___
JOB #: SITE MANAGER:	DATE: H&S COORDINATOR:	SITE LOCATION:
SITE CONDITIONS TEMPERATURE: RELATIVE HUMIDITY: WIND SPEED & DIRECTION:	SITE PERSONNEL:	

SAMPLE NO.	TIME	SAMPLE DESCRIPTION	LOCATION	INSTRUMENT	READING	COMMENTS

OGDEN

SAMPLE No.	TIME	SAMPLE DESCRIPTION	LOCATION	INSTRUMENT	READING	COMMENTS

OGDEN

TITLE FIELD INSTRUMENTATION: THERMO ENVIRONMENTAL INSTRUMENTS INC. OVM MODEL 580B CALIBRATION AND OPERATION

1.0 PURPOSE

This procedure is intended to provide general operational guidelines for the Thermo Environmental Instruments Inc. organic vapor meter 580B. The 580B detects and quantitates most organic vapors with a photoionization detector. The 580B can detect up to 2000 parts per million (ppm).

2.0 SCOPE

This document applies to all OEES personnel involved in the operation, calibration, and maintenance of the 580B.

3.0 DEFINITIONS

Key Pad - There are seven switches on the key pad which operate the 580B. The switch marked ON/OFF is used to turn the pump and lamp on and off. The switch marked LIGHT will turn on backlighting for the two-line display. The other five switches perform various functions.

LIGHT switch - Illuminates the display.

MODE/STORE switch - Causes the 580B to return to the RUN mode. When the 580B is already in the RUN mode, it causes it to enter the LOG mode.

ON/OFF switch - Toggles the lamp and pump power between on and off.

Power Plug - The power plug is used to run the instrument off of its internal batteries. There is a chain attached to the power plug so that it will not be lost.

Pump - The 580B pump draws the sample into the detector.

Sample Exit Port - The 580B sample is drawn into the detector by a positive displacement pump and then sent back out through the exit port.

Sample Inlet - Sample is drawn into the detector through the sample inlet at the front of the 580B.

SPKR switch - Normally is used to toggle the instrument speaker between on and off.

4.0 RESPONSIBILITIES

4.1 Health and Safety Manager (HSM)

The HSM is responsible for approving instrument procedures for issue and general implementation of this procedure.

4.2 Health and Safety Coordinator (HSC)

The HSC arranges the training of personnel in the use of the instruments covered by this procedure.

4.3 Site Health and Safety Coordinator (SHSC)

The SHSC is responsible for general field implementation of this procedure.

5.0 PROCEDURES

5.1 Operating

Attachment 1 depicts the operating features of the 580B.

Turn the OVM Model 580B on. When the instrument is first turned on the display will indicate that the lamp is not lit. Pressing the ON/OFF switch will tell the microprocessor to turn on the lamp and the pump. If the lamp is not lit after 14 seconds the microprocessor will indicate a "lamp out" condition. (If a lamp out condition is indicated check the seating of the lamp.)

Once the lamp is lit the display will show the concentration (in ppm) on the bottom line.

Turn the lamp and pump off by pressing the ON/OFF switch.

When the instrument is operated in RUN mode, a bar graph will show at the top of the screen to show a rough visual indication of the current concentration (in ppm). Remember, the concentration detected by the instrument are a ppm equivalent as compared to the calibrating gas. When the instrument is operated in the MAX HOLD mode, the top line of the display will indicate the maximum reading and the bottom line will indicate the current concentration. Whenever a new maximum is seen the top line will be updated. The MAX HOLD reading may be reset by pressing the RESET button while in the RUN mode.

Audible indication of concentration can be obtained by pressing the SPKR switch while in the RUN mode. The speaker will generate a "clicking" that will increase in speed as the concentration increases. The speaker rate can be changed by changing the switches located inside the side door. Only one of the four speaker rate switches should be on (in the down position) at any time. The 580B will display an overrange warning if the concentration goes above 2000 ppm. The top line of the display will show "overrange" when this occurs. The overrange warning will continue until the instrument is brought to an area with an organic vapor concentration of less than 20 ppm.

The alarm will sound if the concentration rises above the alarm setting. If the speaker is not activated then the alarm sound will not be heard. See Section 5.4 for the procedure for alarm setting. The alarm will turn off once the concentration drops below the alarm setting.

5.2 Run Mode Selection

The 580B has two run modes, MAX HOLD and CONCENTRATION METER. When the display reads "Conc. Meter" on the top line the bottom line will show "RESET TO CHG" and will alternate every two seconds with "MAX HOLD." In its current setup, the 580B is in the "MAX HOLD" mode. To select the RUN mode, press the RESET switch to cause the 580B to show "MAX HOLD +=USE/-=NO." Press the +/INC switch to select the "MAX HOLD" mode. To select the CONCENTRATION METER NORMAL mode, press the -/CRSR switch. The 580B will then return to the previous screen.

5.3 Average Time Selection

The 580B can be configured to display the average concentration from once a second up to once every four minutes. The display will show "AVERAGE = 0:01 RESET TO CHG." When the RESET switch is pressed the display will show "AVERAGE = 0:01 RESET WHEN DONE." Press the +/INC switch to increment the number above the cursor and the -/CRSR switch to move the cursor. The average time format is M:SS (where M is minutes and S is seconds).

5.4 Alarm Setting

The 580B has an alarm that will sound if the concentration rises above the alarm setting. The alarm will not be heard if the speaker has not been activated. The top line of the display will, however, indicate when there is an alarm condition. The 580B will display the current alarm setting on the top line of the display. The setting may be changed by simultaneously pressing the RESET switch with either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor. The alarm setting is entered in the Parameters mode.

5.5 Lamp Selection

The lamp setting can be changed depending on the lamp selected. Refer to the instruction manual for information on changing this setting.

5.6 Response Factor Setting

The 580B will indicate a 1:1 response ratio if exposed to the same gas as the gas to which it was calibrated. However, if the 580B is exposed to a gas or vapor (or mixture of gases/vapors) that are not the same as the calibration gas, it will not detect at a 1:1 response ratio. When the 580B has been calibrated to the span gas, the response factor is:

$$\frac{\text{known concentration of target gas}}{\text{displayed concentration of target gas (as a ppm-equivalent)}}$$

The current Response Factor Setting will be displayed on the top line of the display. Thus, a response factor may be used in order to relate a particular gas to the calibration gas. Response factors can be input when calibrating the MicroTIP. When computing the concentration the microprocessor multiplies the detected concentration by the response factor and displays the result. If the response factor is 1.0 then the concentration is not changed. The instruction manual provides response factors for common gases that may be encountered.

If the 580B Response Factor Setting is set at 1.0 when using the instrument to detect gases/vapors other than the span gas, then the person reading the display should manually perform the following computation:

Actual airborne concentration of target gas =
displayed concentration x response factor of target gas

5.7 Calibration

To calibrate the 580B, perform the following steps:

1. The 580B will display "RESET TO CALIBRATE." Press the RESET switch to enter the calibration mode.

2. The display will show "RESTORE BACKUP +=YES." The previous calibration information may be restored by pressing the +/INC switch. The 580B will then return to the previous screen. If the backup is not desired, press -/INC to continue the calibration routine.

3. The display will show "ZERO GAS RESET WHEN READY." Introduce the zero gas and press RESET. The 580B will then zero the instrument.

4. The display will then read "MODEL 580B ZEROING."

5. When zeroing is complete the display will show "SPAN PPM=0000." Then enter the span gas concentration by simultaneously pressing the RESET switch and either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor.

6. Once the span gas concentration has been entered the +/INC switch should be pressed.

7. The display will then read "SPAN GAS RESET WHEN READY." Introduce the span gas and then press RESET. The 580B will then calibrate the instrument and display "MODEL 580B CALIBRATING."

8. Once calibration is complete, the 580B will go back to the beginning and display "RESET TO CALIBRATE."

9. If, during the zeroing or calibrating of the 580B, a steady reading was not seen, then the 580B will display "CAL ERROR RESET WHEN READY."

10. Press RESET to return the 580B to zeroing or calibrating (depending on which it came from).

11. When calibration is completed, record the readings on an Instrument Calibration Log (Attachment 2).

5.8 Lamp Cleaning

Occasionally the lamp may need to be removed for cleaning. Refer to the instruction manual for information about lamp removal and insertion.

5.9 Low Battery

The 580B will display a warning when the battery is low. The warning will be a flashing B in the left-hand corner of the bottom line of the display when in the RUN mode.

5.10 Parameters Mode

All of the 580B operating parameters are entered in the Parameters mode. Calibration is also performed within the Parameters mode. To enter the Parameters mode, press the -/CRSR switch from the main menu. Pressing the +/INC switch will advance the 580B to the next selection or setting. Pressing the -/CRSR switch will advance the 580B to the previous selection/setting. Once a setting has been selected, the +/INC and -/CRSR switches will have different functions.

6.0 RECORDS

6.1 PROCEDURE

A copy of each approved procedure and revision shall be retained as a permanent record in accordance with company procedures. Monitoring results will be recorded on the Site Air Surveillance Record (Attachment 3).

6.2 CALIBRATION

Instrument Calibration Logs (Attachment 2) will be maintained in the health and safety portion of the project files and/or on a log with the instrument.

7.0 REFERENCES

Thermo Environmental Instruments Inc. OVM/Datalogger Model 580B Instruction Manual.

8.0 ATTACHMENTS

1. Thermo 580B Operating Features
2. Instrument Calibration Log
3. Site Air Surveillance Record

ATTACHMENT 2

INSTRUMENT CALIBRATION LOG

OGDEN

INSTRUMENT CALIBRATION LOG

Project: ______________________

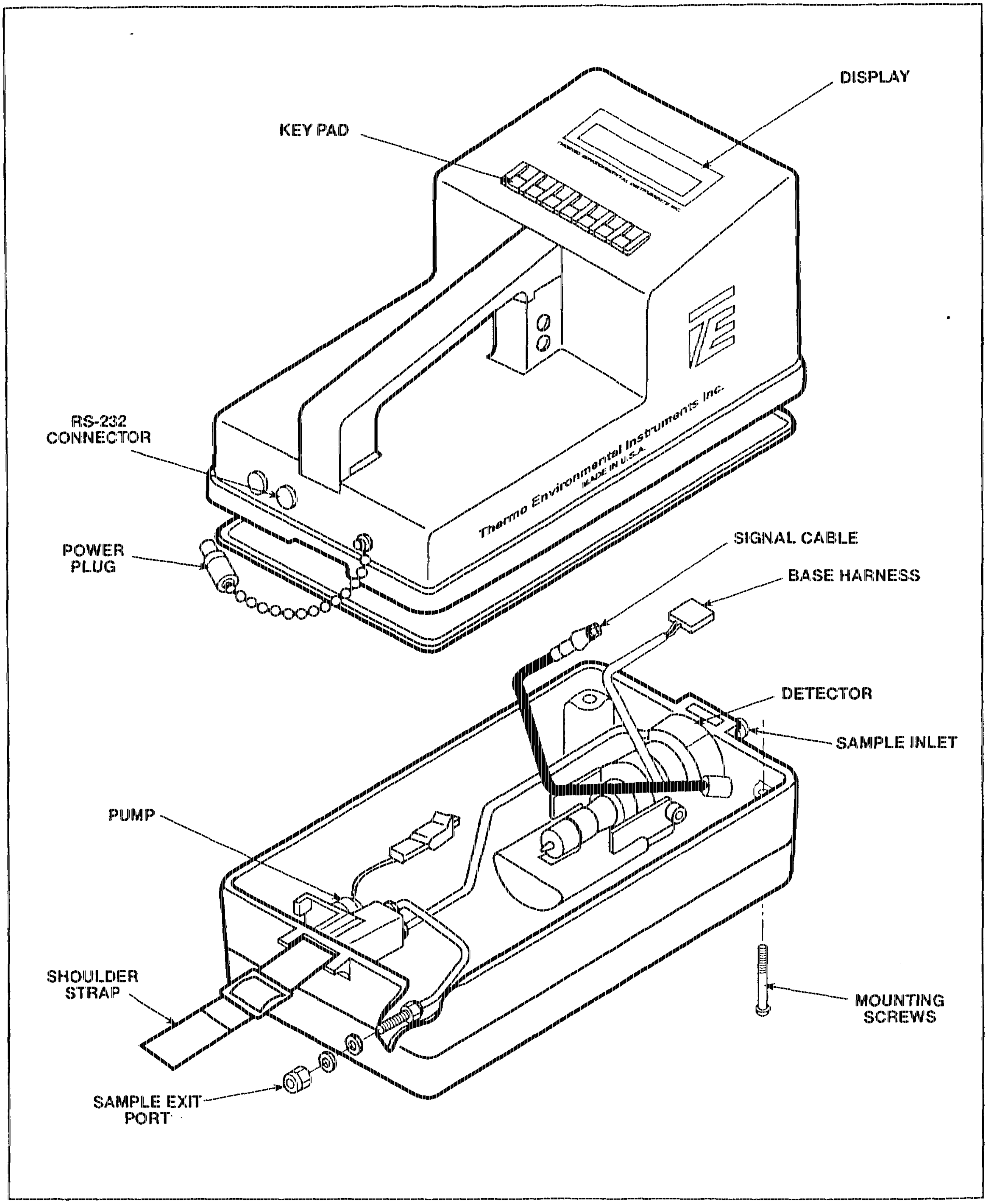
Location: ______________________

JOB #_ ______________________

Date/Time	Instrument	Serial Number	Span Gas Substance	Span Conc.	Instrument Reading at Calibration	Instrument Calibration Reading Post Sampling	Comments

ATTACHMENT 1

THERMO 580B OPERATING FEATURES


DISPLAY
KEY PAD
RS-232
CONNECTOR
Thermo Environmental Instruments Inc.
MADE IN U.S.A.
POWER
PLUG
SIGNAL CABLE
BASE HARNESS
DETECTOR
SAMPLE INLET
PUMP
SHOULDER
STRAP
MOUNTING
SCREWS
SAMPLE EXIT
PORT

ATTACHMENT 3

SITE AIR SURVEILLANCE RECORD

SITE AIR SURVEILLANCE RECORD

SITE INFORMATION JOB #: SITE MANAGER:	DATE: H&S COORDINATOR:	Page ____ of ____ SITE LOCATION:
SITE CONDITIONS TEMPERATURE: RELATIVE HUMIDITY: WIND SPEED & DIRECTION:	SITE PERSONNEL:	

SAMPLE NO.	TIME	SAMPLE DESCRIPTION	LOCATION	INSTRUMENT	READING	COMMENTS

ATTACHMENT E

HOSPITAL ROUTE MAP AND EMERGENCY CONTACTS

EMERGENCY CONTACTS

Emergency resources are as follows:

Local Police Department:	Sauget	618-322-6507/6997
	Cahokia	618-337-5080
Local Fire Department:	Sauget	618-332-6700
	Cahokia	618-337-5080
Local Hospital:	St. Mary's Hospital	
	100 North 8^{th} St.	
	East St. Louis, IL	618-274-1900
Poison Control Center:	1-800-942-5969	
USEPA National Response Center:	800-424-8802	

AMEC Contacts

Ecological Project Manager:	Chuck Harman	732-302-9500, ext. 127
Project Health and Safety Manager:	Jeffrey Tasca	732-302-9500, ext. 106
Corporate Health and Safety Manager:	Denise Daggett, CIH	858-458-9044

Solutia Contact

Project Manager:	Steven Smith	314-674-4922

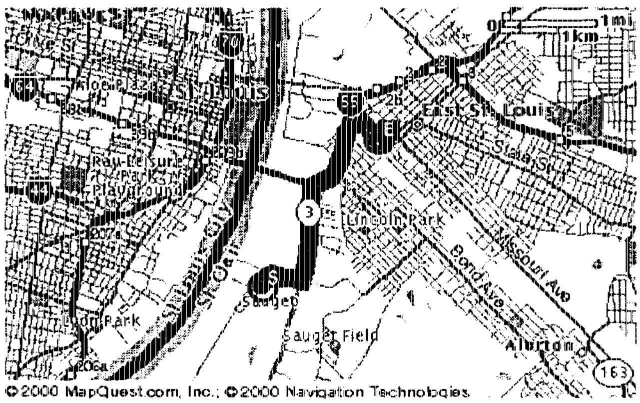
WWW.MAPQUEST.COM

Driving Directions Results

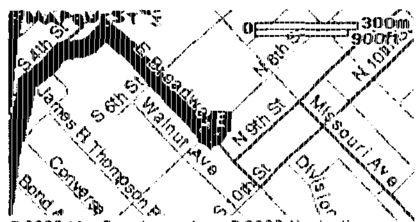
< Back SEND TO PRINTER

FROM:	TO:
MOBILE ST EAST SAINT LOUIS, IL 62201 US	**100 N 8TH ST EAST SAINT LOUIS, IL 62201-2989 US**

DIRECTIONS	DISTANCE
1: Start out going Northeast on MOBILE ST towards MONSANTO AVE by turning right.	0.2 miles (0.3 km)
2: Turn SLIGHT RIGHT onto MONSANTO AVE.	0.3 miles (0.5 km)
3: Turn LEFT onto IL-3.	0.9 miles (1.5 km)
4: Take the I-70 E/I-64 E/I-55 N ramp towards IL-3 N/CHICAGO/INDIANAPOLIS.	0.6 miles (0.9 km)
5: Keep RIGHT at the fork in the ramp.	0.2 miles (0.4 km)
6: Merge onto S 4TH ST.	0.2 miles (0.3 km)
7: Turn RIGHT onto E BROADWAY/IL-15.	0.3 miles (0.4 km)
8: Turn LEFT onto N 8TH ST.	0.0 miles (0.0 km)
TOTAL ESTIMATED TIME: **8 minutes**	**TOTAL DISTANCE:** **2.7 miles** (4.3 km)



TO:
100 N 8TH ST EAST SAINT LOUIS, IL 62201-2989 US



NAVTECH Use Subject to License/Copyright

Coupons Along Your Route

EMERGENCY CONTACTS

Emergency resources are as follows:

Local Police Department:	Sauget	618-322-6507/6997
	Cahokia	618-337-5080
Local Fire Department:	Sauget	618-332-6700
	Cahokia	618-337-5080
Local Hospital:	St. Mary's Hospital	
	100 North 8th St.	
	East St. Louis, IL	618-274-1900
Poison Control Center:	1-800-942-5969	
USEPA National Response Center:	800-424-8802	

AMEC Contacts

Ecological Project Manager:	Chuck Harman	732-302-9500, ext. 127
Project Health and Safety Manager:	Jeffrey Tasca	732-302-9500, ext. 106
Corporate Health and Safety Manager:	Denise Daggett, CIH	858-458-9044

Solutia Contact

Project Manager:	Steven Smith	314-674-4922

ATTACHMENT F

HEALTH AND SAFETY ORIENTATION FORM

HEALTH AND SAFETY ORIENTATION FORM

(Visitors And Non-Routine Subcontractors)

I understand that I am entering a site where hazardous materials have been detected in the environmental media. Chemicals detected include, but are not limited to, polychlorinated biphenyls (PCBs), phenols, anilines, solvents, metals, and polyaromatic hydrocarbons (PAHs). In addition, due to the nature of the work being conducted on the site, physical hazards are present.

The information summarized in this sheet is important for you to read and fully understand. It has been extracted from the Site-specific Health and Safety Plan for the Sauget Area 2 Sites and has been compiled for <u>your</u> health and safety. If you have any questions regarding the information presented below, please ask your escort for identification.

Health, Safety, And Security Information

1. You must sign in and out of the Visitor Log Book maintained at the site. This assists in identifying all personnel at the site in the event of an emergency.

2. You must remain in the support ("clean") zone unless you have an escort. If you are observed alone in any unauthorized area, you will be asked to leave immediately.

3. Areas marked "Caution - DO NOT ENTER" demarcate where the Exclusion ("contaminated") areas begin. You are not authorized to enter these areas.

4. Access to the Exclusion Zone is strictly forbidden to ALL visitors unless they have written approval from the client and proof of adequate OSHA training and medical surveillance PRIOR to arrival on site.

5. Please read and follow all safety signs onsite which will alert you to possible physical and chemical hazards.

6. Eating and smoking are not allowed onsite. You may eat or smoke in designated areas only or in your vehicle.

7. There may be heavy equipment and/or moving vehicles. Please be aware.

8. No one under the age of 18 is permitted onsite without prior written approval from the client.

9. Report any accident or injury to your escort

10. No domestic animals are permitted onsite.

11. Please do not disrupt site activities or contribute to any unnecessary delays.

Emergency Notification

1. in the event of a site emergency, please walk immediately to the designated meeting area for the site. You will receive further instructions at this location. Please stay in this meeting area until the all-clear signal is given from the Field Team Leader or off-site emergency support personnel.

2. Please cooperate fully with those in authority in the event of an emergency.

ACKNOWLEDGEMENT OF INFORMATION

I have read and understand the above information provided by AMEC Earth & Environmental, Inc. and have had an opportunity to direct questions of a health and safety nature and have received adequate answers/explanations from my escort or other site staff member. My signature also indicates that my employer assumes the risk of any injury or property damage that may occur to me or by me.

Print Name	Signature	Affiliation	Date	Time In	Time Out